

Project Report
On
EFFECT OF OXIDIZING AGENTS ON DYEING COTTON
FABRICS WITH VAT DYE

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(This report presented in partial fulfillment of the requirements for the degree of Bachelor of Science in Textile Engineering)

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LETTER OF APPROVAL

November 20, 2014

To

The Head

Department of Textile Engineering

Daffodil International University

102 Sukrabad, Mirpur Road, Dhaka 1207

Subject: Approval of final year project report.

Dear Sir

I am just writing to let you know that, this project report titled as **“Effect of Oxidizing Agents on Dyeing Cotton Fabrics with Vat Dye”** is completed for final evaluation. The whole report is prepared based on proper investigation and interpretation though critical analysis of empirical data with required belongings. The students were directly involved in their project activities and the report becomes vital to spark off many valuable information for the readers.

Therefore, it will highly be appreciated if you kindly accept this project report and consider it for final evaluation.

Yours Sincerely,

Sumon Mazumder

Assistant Professor

Department of TE

FE, DIU

DECLARATION

We hereby declare that, this project has been done by us under the supervision of Sumon Mazumder, Assistant Professor, Department of Textile Engineering, Faculty of Engineering, Daffodil International University. We also declare that, neither this project nor any part of this project has been submitted elsewhere for award of any degree or diploma.

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ABSTRACT

The word vat means, 'Vessel'. The dyes take their name from vatting. The vat dyes are naturally coloring matter and kept in wooden vat and make solubilize vat dyes by the process of fermentation – so it is called vat dyes. Vat dyes are different from reactive dyes, disperse dyes, basic dyes, acid dyes or azoic color but its application process is near similar as sulphur dyes. Vat dyes are used for coloring cellulosic fiber specially cotton fiber. It is widely used for run the dyeing process of cotton yarns which is used for producing jeans or denim product. Vat dyes provide textile material with the best color fastness of all the dyes in common used for cotton fabric. Most vat dyes are found in insoluble oxidized form. The first operation therefore, it requires a reducing agent to solubilize them. This reducing agent converts the dye to its "leuco" form that is soluble. This process commonly referred to as vatting. Once attached to the fabric, the leuco dye is then oxidized to the insoluble state. Two types of oxidation may be done, physical oxidation which is done with atmospheric oxygen and another is chemical oxidation which is carried out with different types of oxidizing agents. Different oxidizing agent gives different color fastness properties. But, the problem is that if the oxidation is done well, the sample will show a bad colorfastness property. We have done a research and it is entitled as "*Effect of Oxidizing Agents on Dyeing Cotton Fabric with Vat Dye*".

During research, we have applied two types of oxidation physical as well as chemical on cotton fabric dyed with vat dye. We have used hydrogen peroxide and sodium per borate as chemical oxidizing agent to analyze the effect on colorfastness properties of eight types of 100% cotton fabric sample like single jersey-140 GSM, single jersey-280 GSM, 2×2 rib, single lacoste, fleece, interlock, slub single jersey which are dyed with vat dye. Then we have gone through some tests like colorfastness to rubbing for dry and wet condition and colorfastness to wash. Finally, we have come to a decision that the colorfastness properties of the samples which are oxidized with chemical oxidation is better than that are oxidized with physical oxidation. Again, among the chemically oxidized samples, the colorfastness properties of the samples which are oxidized with sodium perborate are better than that are oxidized with hydrogen peroxide.

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CHAPTER 01
INTRODUCTION

CHAPTER 01

INTRODUCTION

The vat dye is found amongst the oldest natural coloring matters used for textiles. Indigo has been known in India since the earliest periods of which historical records exist. According to the writings of Julius Caesar the ancient Britons used Woad to stain their Bodies and faces and Tyrian purple was exported from Tyre to the Mediterranean countries nearly 4000 years ago. Tyrian purple is extracted from a shell fish and is therefore of animal origin but woad and indigo exist in plants, combined with glucose in the form of glucosides.

The vat dyes are all insoluble in water and cannot be used for dyeing without modification when treated with reducing agents they are converted into leuco compounds all of which are soluble in water in the presence of alkalis. These leuco compounds are substantive towards cellulose and reoxidize to the insoluble coloured pigment within the fibre when exposed to air. The leuco compounds are often colourless or of quite a different colour from the product of oxidation.

During dyeing process of cotton fabric with vat dye oxidation is occurred. Without occurring oxidation, vat dye may not be insoluble and thus during washing color may be washed away. So it is very important to be oxidised.

We will oxidise the dyed fabric in two ways-one is physical oxidation and another is chemical oxidation. After completing oxidation process we will have some test methods like color fastness to wash or color fastness to rubbing in case of dry and wet phase on fabric. After having these test on the fabric we will make the data for perfect analysis.

CHAPTER 02
LITERATURE REVIEW

CHAPTER 02

LITERATURE REVIEW

2.1 Cotton

Cotton, one of the world's leading agricultural crops, is plentiful and economically produced; making cotton products relatively inexpensive. Using cotton fiber after different processing steps cotton fabric can be produced. Cotton fabrics can be extremely durable and resistant to abrasion. Cotton accepts many dyes, is usually washable, and can be ironed at relatively high temperatures. Cotton fiber is a cellulosic fiber. It has worldwide popularity for its variety of use, cotton has its own physical and chemical properties which are required to know for better processing in spinning, weaving, knitting, dyeing, printing as well as finishing. Among all the textile fabrics cotton fabrics are more comfortable to wear in all season [1]



Figure 2.1: Cotton fiber tree

Characteristics of Cotton

Cotton, as a natural cellulosic fiber, has a lot of characteristics, such as;

- Comfortable Soft hand
- Good absorbency
- Color retention

- Prints well
- Machine-washable
- Dry-cleanable
- Good strength
- Drapes well
- Easy to handle and sew [2]

2.1.1 Fiber structure and formation

The botanical name of American Upland cotton is *Gossypium Hirsutum* .

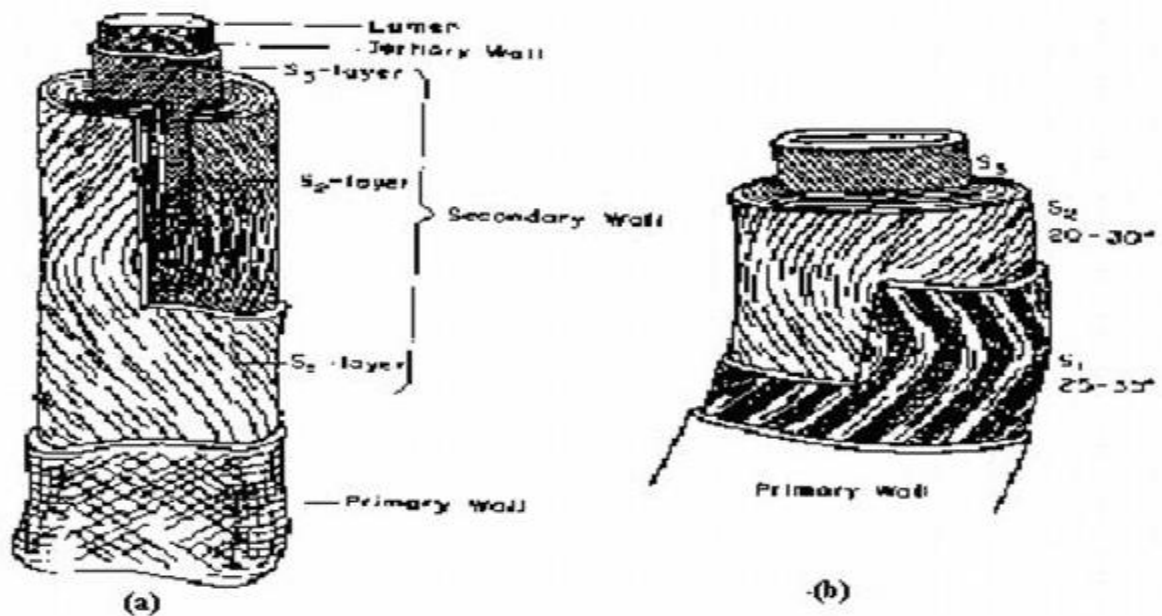


Figure 2.2: Schematic diagram of cotton fiber

Each cotton fiber is composed of concentric layers. These are as follows.....

The cuticle layer:

The cuticle layer on the fiber itself is separable from the fiber and consists of wax and pectin materials.

The primary wall:

The primary wall, the most peripheral layer of the fiber, is composed of cellulosic crystalline fibrils.

The secondary wall:

The secondary wall of the fiber consists of three distinct layers. All three layers of the secondary wall include closely packed parallel fibrils with spiral winding of 25-35° and represent the majority of cellulose within the fiber.

Lumen:

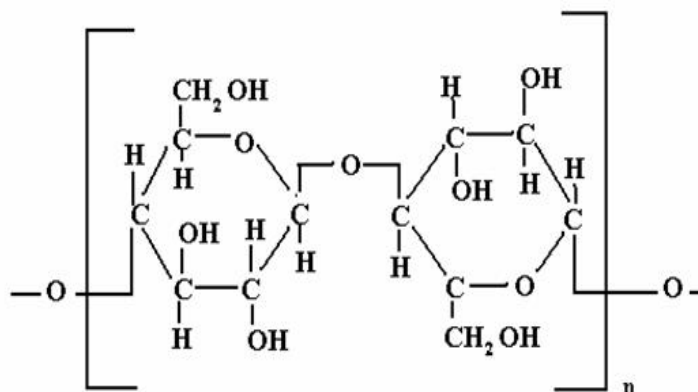
The innermost part of cotton fiber- the lumen- is composed of the remains of the cell contents. Before boll opening, the lumen is filled with liquid containing the cell nucleus and protoplasm.

Cross section:

The cross section of the fiber is bean-shaped, swelling almost round when moisture absorption takes place. [2]

Repeat Unit Of Cellulose:

The repeat unit of cellulose contains hydroxyl group(-OH).The current consensus regarding cellulose crystallinity is that fibers are essentially 100% crystalline and that very small crystalline units imperfectly packed together cause the observed disorder.[2]



Morphology Of The Cotton Plant

Roots

Cotton grows an extensive root system that generally reaches at least 1 yard deep. Cotton grown in quality soil also features long lateral roots.

Stem

The stem of a cotton plant extends vertically from the root and gives way to several branches. Stems are topped by bulbous cotton bolls.

Fertilization

The cotton plants are self-fertilizing, without the use of wind. Insects may contribute to cross pollination.

Boll

The fruit of a cotton plant is known as a boll, according to the Ikisan website. Bolls usually divide into four to six compartments, and each one contains many seeds. Long fibers of cotton plant surround the fruit.

Fiber Quality

According to the Clemex website, the quality of cotton fibers is determined by taking samples from ginning fibers. A fiber's length, strength cleanliness and color are evaluated.
[3]

Macro Structure Of Cotton Fibers

- Length : 1 cm to 6.5 cm
- Diameter: 11 um to 22 um
- Convolutions : Sixty per centimeter. (Creamy or brown.)
- Color : Generally White, may be Creamy or Brown.
- Length:Width: 6000:1
- Light reflection: Low lusture, dull.[4]

Cultivation Of Cotton Plant

The cotton plant can be found as a perennial in treelike plants in tropical climates but is normally cultivated as a shrubby annual in temperate climates. Whereas it grows up to 6 metres (20 feet) high in the tropics, it characteristically ranges from 1 to 2 metres (3 to 6.5 feet) in height under cultivation.

Within 80–100 days after planting, the plant develops white blossoms, which change to a reddish colour. The blossoms fall off after a few days and are replaced by small green triangular pods, called boll that mature after a period of 55–80 days.

During this period the seeds and their attached hairs develop within the boll, which increases considerably in size. The seed hair, or cotton fibre, reaching a maximum length of about 6 cm (2.5 inches) in long-fibre varieties, is known as lint. Linters, fibres considerably shorter than the seed hair and more closely connected to the seed, come from a second growth beginning about 10 days after the first seed hairs begin to develop.

When ripe, the boll bursts into a white, fluffy ball containing three to five cells, each having 7 to 10 seeds embedded in a mass of seed fibres. Two-thirds of the weight of the seed cotton (i.e., the seed with the adhering seed hair) consists of the seeds. The fibres are composed of about 87 to 90 percent cellulose (a carbohydrate plant substance), 5 to 8 percent water, and 4 to 6 percent natural impurities.[5]

2.1.2 Physical Properties Of Cotton

Fiber Length

Fiber length is described as "the average length of the longer one-half of the fibers. Typical lengths of Upland cottons might range from 0.79 to 1.36 inch. Cottons come from the cotton plant; the longer strand types such as Pima or Sea Island produce the finest types of cotton fabrics .

Length Uniformity

Length uniformity or uniformity ratio is determined as " a ratio between the mean length and the upper half mean length of the fibers and is expressed as a percentage". Typical comparisons are illustrated below.

Table 2.1: Length uniformity and their index.

LENGTH UNIFORMITY	UNIFORMITY INDEX [%]
Very High	>85
High	83-85
Intermediate	80-82
Low	77-79
Very Low	<77

Fiber Strength

Fiber strength is measured in grams per denier. It is determined as the force necessary to break the beard of fibers, clamped in two sets of jaws, (1/8 inch apart).

The breaking strength of cotton is about 3.0~4.9 g/denier.

The breaking elongation is about 8~10%.

Typical tensile levels are illustrated.

Table No.2.2: Degree of strength and fiber strength

DEGREE OF STRENGTH	FIBER STRENGTH [g/tex]
Very Strong	>31
Strong	29-30
Average	26-28
Intermediate	24-25
Weak	<23

Micronaire

Micronaire measurements reflect fiber fineness and maturity. Typical micronaire levels are illustrated as belows.

Table No.2.3: Cotton range and micronaire readings

COTTON RANGE	MICRONAIRE READING
Premium	3.7-4.2
Base Range	4.3-4.9
Discount Range	>5.0

Influence of micronaire on nep formation during processing of cotton has been given below, [6]

Table No.2.4: Influence of micronaire on nep formation

Property	High Micronaire	Low Micronaire
Micronaire	4.5	3.0
Length (inches)	1.0	1.0
Length uniformity (%)	79.4	78.8
Strength (g/tex)	25.0	25.0
Elongation (%)	7.0	7.4
Neps per gram:	-	-
Original bale	375	574
Mechanically cleaned	354	860
Bleached	520	1140
Card web	540	2406

Color

The color of cotton samples is determined from two parameters: degree of reflectance (Rd) and yellowness (+b). Degree of reflectance shows the brightness of the sample and yellowness depicts the degree of cotton

Leaf Grade

Leaf grade is provided visually as the amount of cotton plant particles within the sample. There are seven leaf grades (1-7) and one below grade (8).

Preparation

Preparation is the classer's interpretation of fiber process ability in terms of degree of roughness or smoothness of ginned cotton.

Neps

A nep is a small tangled fiber knot often caused by processing. Neps can be measured by the AFIS nep tester and reported as the total number of neps per 0.5 gram of the fiber and average size in millimeters.[2]

Elongation At Break

Cotton does not stress easily. It has an elongation at break of 5-10%.

Elastic Recovery

Cotton is inelastic and rigid fiber. At 2% extension it has an ER of 74% and 5% extension it has an ER of 45%.

Specific Gravity

Its specific gravity is 1.54.

Moisture Regain(MR%)

Standard moisture regain is 8.5%.[1]

2.1.3 Chemical Properties Of Cotton

Cotton is a natural cellulosic fiber and it has some chemical properties. Chemical properties of cotton fiber are given below.

Effect Of Acids

Cotton is attacked by hot dilute acids or cold concentrated acids which it disintegrates. It is not affected by acid.

Effect Of Alkalis

Cotton has an excellent resistance to alkalis. It swells in caustic alkalis (NaOH) but it does not damage. It can be washed repeatedly in soap solution without any problem.

Effect Of Organic Solvent

Cotton has high resistance to normal cleaning solvents. Cotton is dissolved by the copper complexes, such as cuprammonium hydroxide, cupriethylene diamine and concentrated 70% H₂SO₄.

Effect Of Insects

Cotton is not attacked by moth-grubs or beetles.

Effect Of Micro Organism

Cotton is attacked by fungi and bacteria. Mildews will feed on cotton fabric, rotting and weakening the materials. Mildews and bacteria will flourish on cotton under hot and humid condition. [1]

Degree Of Polymerization And Crystallinity Of Cellulose Fiber

Degree of polymerization and crystallinity of cellulose fibers are given below,

Table No.2.5: Degree of polymerization and crystallinity of cellulose fibers

Fiber	Average Degree of Polymerization (DP)	Average Crystallinity (%)
Cotton	9,000–15,000	73
Viscose rayon (Regular)	250–450	60
High tenacity	500–650	-
High wet modulus	400–550	-
Wood pulp	600–1,500	35

2.1.4 Uses Of Cotton Fiber

Cotton fiber is a versatile fibre which has wide variety of uses. But the Cotton fibre is mostly used on the Apparel Industry to make the wearing cloth like Sweaters, Skirts, Shirts, Swimwear, Kids wear, Blouses, Pants, Hosiery and to make other type of dresses.[7]

2.2. Oxidizing Agent

An oxidizing agent can be defined as a substance that removes electrons from another reactant in a redox chemical reaction. The oxidizing agent is "reduced" by taking electrons onto itself and the reactant is "oxidized" by having its electrons taken away. Oxygen is the prime example of an oxidizing agent, but it is only one among many.

On the other hand we defined it as “An oxidizing agent, or oxidant, gains electrons and is reduced in a chemical reaction. Also known as the electron acceptor, the oxidizing agent is normally in one of its higher possible oxidation states because it will gain electrons and be reduced. Examples of oxidizing agents include halogens, potassium nitrate, and nitric acid.”

We also defined it as an oxidizing agent is referred to as a chemical compound that readily transfers oxygen atoms or a substance that gains electrons in a redox chemical reaction.

In simple terms:

- The oxidizing agent takes electrons from another species, and thus itself is reduced.
- The reducing agent gives electrons to another species, and thus itself is oxidized.
- All atoms in a molecule can be assigned an oxidation number. This number changes when an oxidant acts on a substrate.
- Redox reactions occur when oxidation states of the reactants change.

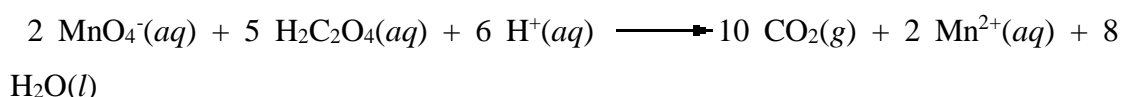
Common Oxidizing Agents

- Oxygen (O_2)
- Ozone (O_3)
- Hydrogen peroxide (H_2O_2) and other inorganic peroxides
- Fluorine (F_2), chlorine (Cl_2), and other halogens

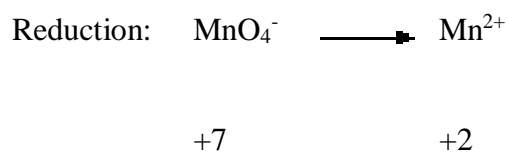
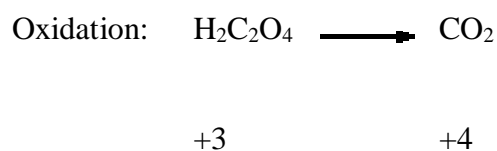
- Nitric acid (HNO₃) and nitrate compounds
- Sulfuric acid (H₂SO₄)
- Peroxydisulfuric acid (H₂S₂O₈)
- Peroxymonosulfuric acid (H₂SO₅)
- Chlorite, chlorate, perchlorate, and other analogous halogen compounds
- Hypochlorite and other hypohalite compounds, including household bleach (NaClO)
- Hexavalent chromium compounds such as chromic and dichromic acids and chromium trioxide, pyridinium chlorochromate (PCC), and chromate/dichromate compounds
- Permanganate compounds such as potassium permanganate
- Sodium perborate
- Nitrous oxide (N₂O)
- Silver oxide (Ag₂O)
- Osmium tetroxide (OsO₄)

2.2.1 Chemistry Of Oxidizing Agents

- In looking at oxidation-reduction reactions, we can focus on the role played by a particular reactant in a chemical reaction.



- Oxalic acid is oxidized to carbon dioxide in this reaction and the permanganate ion is reduced to the Mn²⁺ ion.

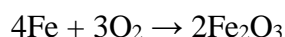


- The permanganate ion removes electrons from oxalic acid molecules and thereby oxidizes the oxalic acid.

Oxidation

Oxidation is defined in chemistry as gain of oxygen, loss of hydrogen or loss of electrons; the loss of electrons enables you to calculate an oxidation state. Example:

The formation of iron(III) oxide;



In the above equation, the iron (Fe) has an oxidation number of 0 before and 3+ after the reaction. For oxygen (O) the oxidation number began as 0 and decreased to 2-. These changes can be viewed as two "half-reactions" that occur concurrently:

1. Oxidation half reaction: $\text{Fe}_0 \rightarrow \text{Fe}_{3+} + 3\text{e}^-$
2. Reduction half reaction: $\text{O}_2 + 4\text{e}^- \rightarrow 2 \text{O}_{2-}$

Iron (Fe) has become oxidized because its oxidation number increased and was the reducing agent because it gave electrons to the oxygen (O). Oxygen (O) has been reduced because the oxidation number has decreased and is the oxidizing agent because it took electrons from iron (Fe).[8]

2.2.2 Properties And Use Of Some Oxidizing Agent

Chromium Reagents (Chromate):

All forms of Cr(VI) are powerful oxidizing agents, and oxidize any CH bonds on a carbon with an oxygen as far as possible without breaking any carbon-carbon bonds; for example, secondary alcohols are converted to ketones, and aldehydes to carboxylic acids.

The most common reagents are: H_2CrO_4 ; $\text{K}_2\text{Cr}_2\text{O}_7 + \text{H}_2\text{SO}_4$; $\text{CrO}_3 + \text{H}_2\text{SO}_4$; they are approximately equivalent. They oxidize the activated CH bonds next to an aromatic ring, the "benzyl" hydrogens, so completely that they usually convert any alkyl benzene to a benzoic acid.

Cr(VI) reagents are so powerful that they can also oxidize alkenes and alkynes, breaking the carbon-carbon bond as ozone does, but this reaction is not synthetically useful.

In non-aqueous solutions, oxidation by Cr(VI) does not go to completion thus, under these conditions, primary alcohols may be oxidized to aldehydes without forming carboxylic acid. The most common reagents for this partial oxidation are: PCC, or pyridinium chlorochromate (formed by dissolving CrO_3 and HCl in pyridine); Collins reagent (CrO_3 in CH_2Cl_2); chromyl chloride (CrO_2Cl_2).

Cr(VI) reagents have been shown to be carcinogenic, upon ingestion either through the stomach or the lungs.

Manganese Reagents (Permanganate):

Permanganate ion, KMnO_4 , will accomplish many of the reactions that chromate will. It is most commonly used in basic solution where it is reduced to the brownish-black solid, MnO_2 ; in acid solution, it is reduced to the pink, soluble ion Mn^{++} . With heat in base, it oxidizes alcohols without breaking carbon-carbon bonds and benzyl CH bonds completely to benzoic acids (their salts since the solution is basic).

There are no deactivated forms of permanganate that permit oxidation of primary alcohols to aldehydes. However, careful reaction of cold basic permanganate with alkenes will give 1,2-diols stereospecifically as osmium tetroxide does. Heating that mixture will cause cleavage of the CC bond.

Osmium Tetroxide:

Another extremely toxic compound, OsO_4 , is used to selectively and stereospecifically oxidize alkenes to 1,2-diols. The reaction is usually done with another oxidizing agent in the solution to regenerate the OsO_4 so that only a catalytic amount of the reagent need be used. Its toxicity is greater than lead or mercury, and similar in effect, attacking the nervous system and the liver.

Sodium Metaperiodate:

Sodium metaperiodate and lead tetracetate also oxidize 1,2-diols, but they cleave the carbon-carbon bond between them like hot basic permanganate.

Nitric Acid:

Concentrated nitric acid is a 69% aqueous solution, but is extremely dangerous because it is a rapid and powerful oxidizing agent. For example (do not try this except in a fume hood and with supervision), it reacts with copper to form greenish cupric nitrate in

solution and gaseous oxides of nitrogen which include NO_2 , the brown gas which makes photochemical air pollution so corrosive to tissues.

In the presence of sulfuric acid it forms NO_2^+ which adds to aromatic rings; since they subsequently lose a hydrogen, the overall reaction is oxidation. Some of the resulting products, such as TNT and TNB, are explosive: the high concentration of oxygen in the molecule provides an internal source of oxygen for very rapid oxidation. (An explosion is a redox reaction that propagates so fast that the heat cannot be dissipated.)

Concentrated nitric acid will convert alcohols to nitrate esters. Although the esterification is not an oxidation, the products of that reaction can also be explosives - such as nitroglycerin and nitrocellulose.

Nitrous Acids:

Nitrous acid, HNO_2 , can be used (with sulfuric acid as a catalyst) to put an NO (nitroso) substituent on an aromatic ring; this nitrosation reaction is exactly analogous to the nitration reaction. But nitrous acid is far more famous for its ability to oxidize amines and amides to N-nitroso compounds ($\text{R}_2\text{-N-N=O}$).

With anilines, the end product is a diazonium salt which can be used to make azo dyes or, with N_2 as a leaving group, to attach nucleophiles to the aromatic ring.

With other amines and amides, the resulting N-nitroso compounds are quite carcinogenic and can be formed in meats that have been cured with sodium nitrite, the salt of nitrous acid. There is no doubt that cultures using a lot of nitrate- and nitrite-cured meats have a higher incidence of cancer of the digestive tract.

Ozone:

Ozone, O_3 , is bent and polar. It is made in the upper atmosphere by collision of an oxygen atom with an oxygen molecule and in the lower atmosphere by reaction of oxygen with oxides of nitrogen formed in photochemical smog. Its polarity makes it fairly reactive.

One common reaction is an addition to alkenes to form a five-membered ring, with the three oxygens still attached to one another called a molozonide, which rearranges and ultimately converts the carbon-carbon double bond into two carbon-oxygen double bonds.

If the reaction is done in the presence of oxidizing agents (like hydrogen peroxide, below), carboxylic acids and ketones are formed, but it is possible to collect aldehydes (and ketones) if the reaction is done in the presence of zinc.

Oxygen:

Dioxygen, O_2 , is rarely used as a reagent in a laboratory setting, but is commonly used in the chemical industry, where cost is the most important thing. Usually a catalyst is needed and/or heat, or even better *light*. It is UV light that makes it possible for dioxygen to dissociate to form oxygen atoms, which react with dioxygen to form ozone; lower energy light will produce an excited state of oxygen which reacts with alkenes.

Even without light, oxygen will slowly oxidize aldehydes when they are exposed to air (to the carboxylic acid), but most reactions with oxygen at the concentration of the atmosphere are too slow at room temperature to be noticed.

The small amount of reaction of oxygen with ethers produces peroxides which can detonate with the slightest touch; anhydrous ethers should be handled and used up quickly to minimize the chance of this happening

Hydrogen Peroxide:

Hydrogen peroxide, H_2O_2 , is a moderately strong oxidizing agent; it even slowly oxidizes water. Its reactions tend to occur via radical paths, but it is easy to understand the reactivity if you imagine the HO_2H to split into OH^- and OH^+ ; the OH^+ would of course be the oxidizing agent, as it desperately needs electrons. Its reactivity increases if the peroxy (OOH) part of the molecule is linked to an electron withdrawing group, as in peroxyacids.

Peroxyacids:

Peroxyacids (also known as peracids), RCO_3H , have an OOH group attached to the carbonyl group. It is even more helpful here to think of the actual oxidizing agent as OH^+ , since the RCO_2^- is such a good leaving group. Peroxyacids are stronger oxidizing agents than hydrogen peroxide itself, converting alkenes into epoxides, for example. This reaction with alkenes is mechanistically similar to the addition of bromine to alkenes; the bromonium ion intermediate looks a lot like epoxide (except for the charge).

Halogens:

In oxidizing ability, the halogens follow the expected order: $F_2 > Cl_2 > Br_2 > I_2$. All should be treated with great respect when working with them, as they can do a great deal of tissue damage very quickly.

Fluorine reacts so explosively with so many organic compounds that working with it requires special facilities; most people send things out for fluorine treatment to the experts.

The most commonly used halogens are Cl_2 and Br_2 ; Cl_2 , a gas, can be generated in dilute solution from bleach, $NaOCl$ (more about this later) and Br_2 can be purchased - it is a volatile reddish-brown corrosive liquid.

Chlorine and bromine atoms (radicals) formed from organohalogen compounds that make it to the stratosphere are responsible for the partial destruction of the ozone layer; they catalyze the conversion of O_3 back to O_2 .

In addition, halogens undergo polar reactions. For example, they add to the carbon-carbon pi bond of alkenes and alkynes and the halogenate very rapidly the alpha-CH bond of aldehydes, ketones and carboxylic acid esters .

Weak Oxidizing Agents:

Very mild oxidizing agents such Ag^+ , Cu^{++} are usually used to do easy oxidations, such as oxidation of an aldehyde to a carboxylic acid in the presence of other oxidizable groups such as alcohols. Both are used to test for reducing sugars (i.e. oxidizable sugars which can reduce the reagent), which all have aldehyde groups.

Tollens' reagent consists of a solution of $Ag(NH_3)_2^+$ which forms a silver mirror on the glass when reduced. Fehling's and Benedict's tests consist of blue Cu^{++} complexed with tartrate or citrate respectively; both produce a brick-red precipitate of Cu_2O upon reduction. [9]

2.2.3 Use Of Oxidizing Agents In Textile Wet Processing

1)Desizing:

Desizing is the process of removing the starch or size covering the warp yarn using enzyme. Oxidizing agents or other chemicals lastly is oxidative desizing method. This method using hydrogen peroxide and persulphate that will degrade the starch with serious attack.

2)Oxidized Starch:

The starch derivatives made by oxidative processes find limited use in textile sizing because they are more expensive to produce as compared to the other starch modifications which can give essentially similar properties to high solids/low viscosity cooks. Instead of using acid to reduce the starch chain length, sodium hypochlorite, an oxidizing agent, is employed.

3) Bleaching:

Cotton being a vegetable fibre will be bleached using an oxidizing agent Bleaching is a process of destruction of natural coloring matter to produce white fabric and be accomplished with a minimum damage to the cloth being bleached. Cotton being a vegetable fiber will be bleached using an oxidizing agent, such as dilute sodium hydrochloride or dilute hydrogen peroxide. If the fabric is to be dyed a deep shade, then lower levels of bleaching are acceptable. [10]

2.3.Vat Dye

Vat dyes are an ancient class of dyes, based on the natural dye, indigo, which is now produced synthetically. Modern vat dyes (1901) include anthraquinone derivatives, but indigo remains the most important vat dye. They are useful mainly for cotton; but also for wool and other fibres.

Cotton, wool, and other fibers can be all dyed with vat dyes. Although almost all dyeing can be done in a vat, the term vat dye is used to describe a chemical class of dyes that are applied to cellulosic fibre (i.e cotton) using a redox reaction as described below. Because of the use of caustic soda, and the very high ph of the dye bath in the dyeing process, wool cannot be dyed using vat dyestuffs. Wool is soluble in caustic soda solutions.[11]

Why Is So Called Vat Dye

The word vat means, 'Vessel'. The dyes take their name from vatting. The vat dyes are naturally coloring matter and kept in wooden vat and make solubilise vat dyes by the process of formantation – so it is called vat dyes. They are applied in a special kind of a dyebath in which the dye is reduced to a soluble form by means of a strong reducing agent, such as hydrosulphite. The vat dyes are insoluble and cannot be used directly and requires vatting. Among all the dyes, it has the best fastness properties.[12]

History Of Vat Dye

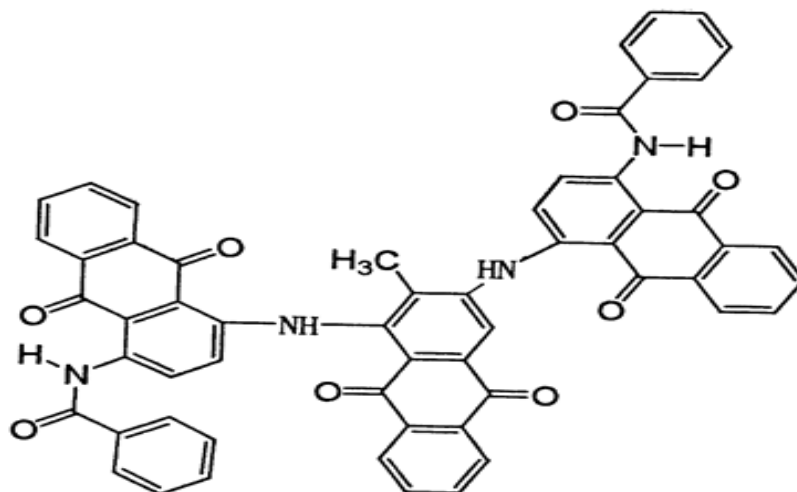
Vat dye is found amongst the oldest natural coloring matters used for textiles. Indigo has been known in India since the earliest periods of which historical records exist. According to the writings of Julius Caesar the ancient Britons used woad to stain their Bodies and faces and Tyrian purple was exported from Tyre to the Mediterranean countries nearly 4000 years ago. Tyrian purple is extracted from a shell fish and is therefore of animal origin but woad and indigo exist in plants, combined with glucose in the form of glucosides the vat dyes are all insoluble in water and cannot be used for dyeing without modification when treated with reducing agents they are converted into leuco compounds all of which are soluble in water in the presence of alkalis. These leuco compounds are substantive towards cellulose and re oxidize to the insoluble coloured pigment within the fibre when exposed to air. The leuco compounds are often colourless or of quite a different color from the product of oxidation.

Vat dyes so named: the word vat means vessel. The dye takes their generic name from vatting , the vat dyes are naturally obtained colouring matter from the ancient time and kept into wooden vat and make solubilise in vat by the process of fermentation – so it is called vat dye.

2.3.1 Chemical Structure,Manufacturing Methods And Their Properties Of Vat Dye

1.Name: C.I .Vat Black 30,C.I.650220

Molecular Structure: anthraquinones



Molecular Formula: C₅₇H₃₄N₄O₈

Molecular Weight: 902.90

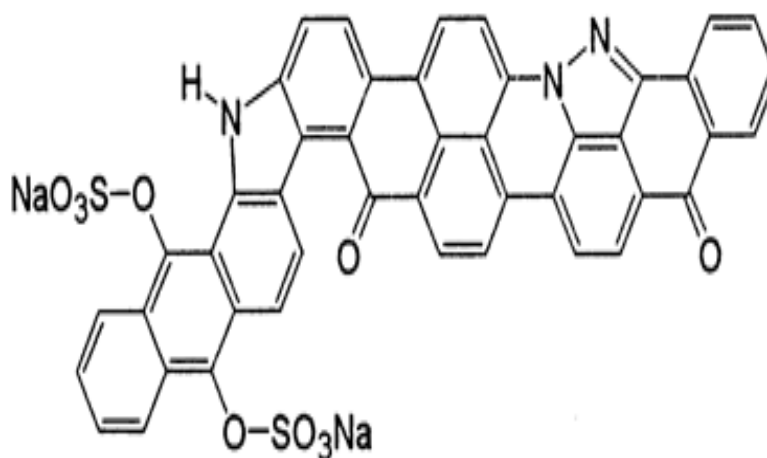
CAS Registry Number: 61902-44-1

Properties and Applications: red light grey.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	4	4	7	4	4-5	4-5	5

2.Name: C.I. Solubilised Vat Black 8, C.I.71001

Molecular Structure: anthraquinones



Molecular Weight: 871.76

CAS Registry Number: 34599-04-7

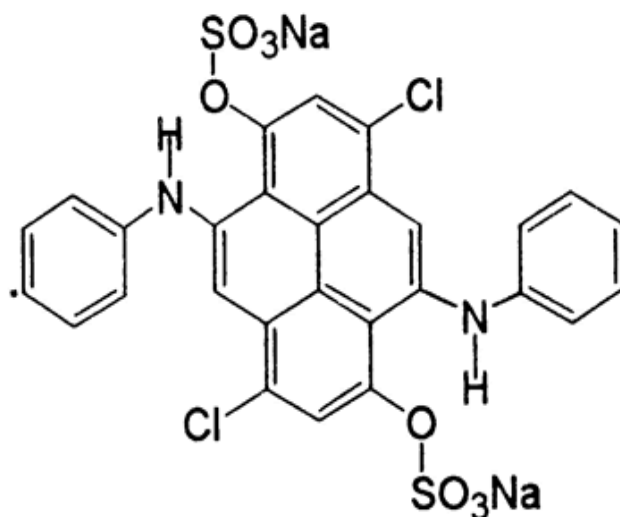
Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Black 8 leuco; (b) in the Pyridine with metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl chlorosulfonate, etc.) processing C.I. Vat Black 8. The above two kinds of situation after alkaline, filter out insolubles, remove Pyridine and salting out.

Properties and Applications: blue light grey.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	4	4-5	7	4	5	4	5

3.Name: C.I .Solubilised Vat Green 21, C.I.59051

Molecular Structure: Pyrene class



Molecular Formula: C₂₈H₁₆Cl₂N₂Na₂O₈S₂

Molecular Weight: 689.45

CAS Registry Number: 6054-59-7

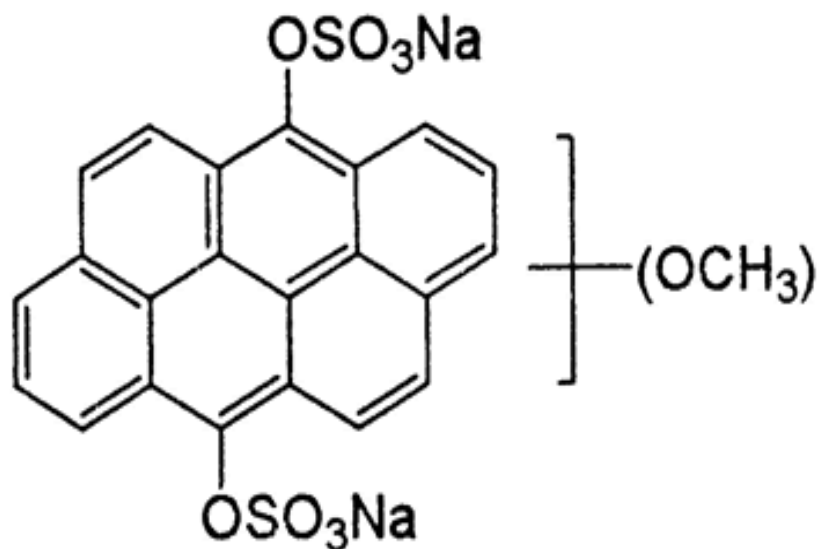
Manufacturing Methods : see soluble VAT dye general methods namely: (a) in the presence of Pyridine , with Chlorosulfonic acid treatment VAT dye (C.I. 5905) leuco; (b) VAT dye (C.I. 5905) in Pyridine with metal (such as copper, iron or cast) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl chlorosulfonate, etc.) processing. Above two kinds of circumstances are alkaline, filter out insolubles, remove Pyridine and salting out.

Properties and Applications: green

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO		5	7-8				

4.Name: C.I. Solubilised Vat Violet 6, C.I. 59316

Molecular Structure: anthraquinones



Molecular Formula: C₂₄H₁₄Na₂O₁₀S₂

Molecular Weight: 572.47

CAS Registry Number: 1324-18-1

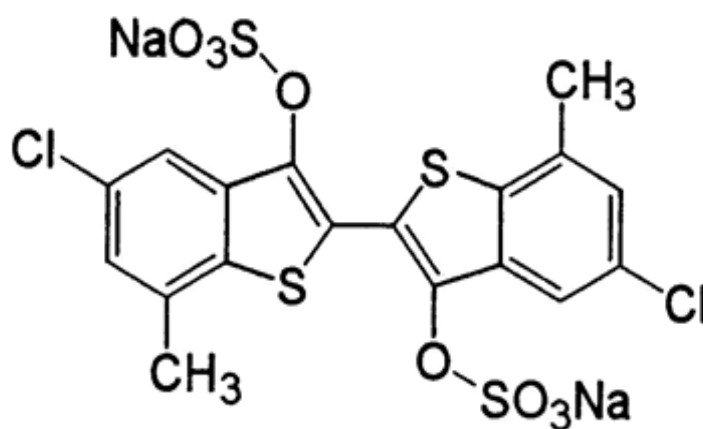
Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Violet 6, leuco; (b) in the Pyridine with metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl chlorosulfonate, etc.) processing C.I. Vat Violet 6. The above two kinds of situation after alkaline, filter out insolubles, remove Pyridine and salting out.

Properties and Applications: red light purple.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	4-5	4-5	7	4-5	5		

5.Name: C.I. Solubilised Vat Violet 2, C.I.73386

Molecular Structure: Thioindigo class



Molecular Formula: C₁₈H₁₂Cl₂Na₂O₈S₄

Molecular Weight: 599.42

CAS Registry Number: 10126-91-7

Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Violet 2 leuco; (b) in the Pyridine with metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or

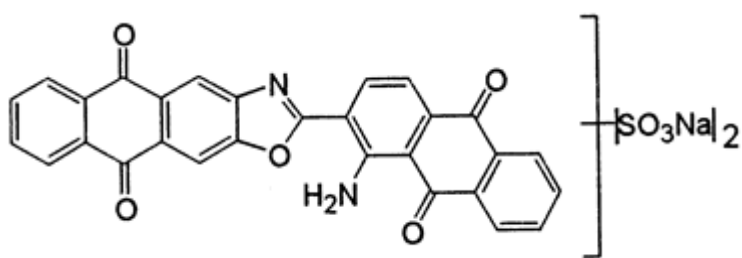
Methyl chlorosulfonate, etc.) processing C.I. Vat Violet 2. The above two kinds of situation after alkaline, filter out insolubles, remove Pyridine and salting out.

Properties and Applications: brilliant red light purple. Purple powder. Soluble in water for red light purple, phenolphthalein is alkaline, insoluble in Ethanol and organic solvent, in light and acid under the action of not easy decomposition. Used for dyeing cotton fabric for brilliant red light purple. In neutral or weak alkaline dyeing bath dyeing. Can be used for cotton fabric of direct, resist, white discharge printing, also can be used for silk and wool dyeing.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	5	5	5	4	4	4	5
AATCC	4	5	5	3-4	4		

6.Name: C.I. Solubilised Vat Red 10,C.I.67001

Molecular Structure: anthraquinones



Molecular Formula: C₂₉H₁₆N₂Na₂O₁₁S₂

Molecular Weight: 678.56

CAS Registry Number: 10126-90-6

Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Red 10 leuco ; (b) used in Pyridine metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl

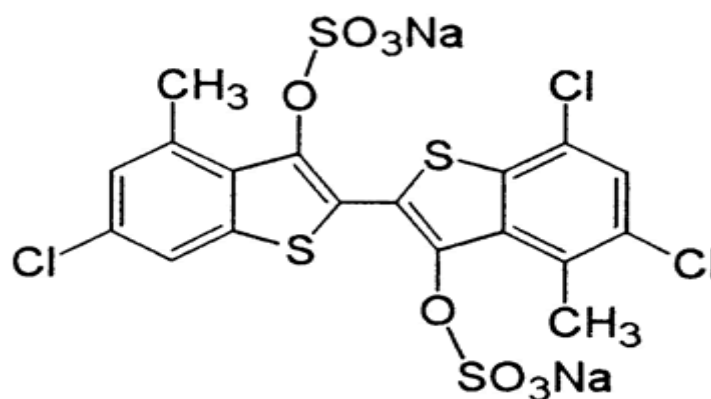
chlorosulfonate) processing C.I. Vat Red 10. The above two kinds of circumstances are through with alkali, filter out the insoluble, remove Pyridine and salting-out.

Properties and Applications: blue-ray red. Red brown powder. Soluble in water, solubility is better. Used for the cotton fabric dyeing as blue-ray red. Dye with neuter or small when cotton with alkaline bath, also can be used for direct printing, the cotton fabric dye printing, discharge printing. Also can be used for silk and wool dyeing and polyester/cotton blended fabric dyeing.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	3	5	6-7	4	4-5	5	5
AATCC	4	5	6	4	4-5		

7.Name: C.I. Solubilised Vat Red 2,C.I.73366

Molecular Structure: Thioindigo class



Molecular Formula: $C_{18}H_9Cl_3Na_2O_8S_4$

Molecular Weight: 633.86

CAS Registry Number: 10126-97-3

Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Red 2 leuco ; (b) used in Pyridine metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl

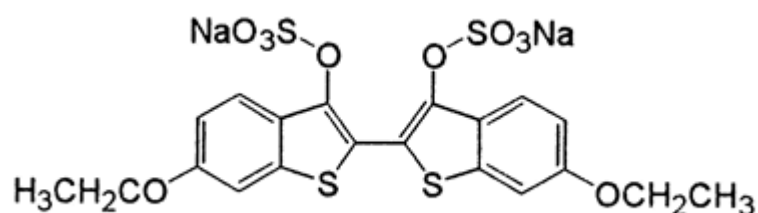
chlorosulfonate) processing C.I. Vat Red 2. The above two kinds of circumstances are through with alkali, filter out the insoluble,, remove Pyridine and salting-out.

Properties and Applications: bright blue light pink to red colourful blue-ray. Shallow red powder. Soluble in water for blue-ray red, solubility in general. This product has the brittle light. Used for the cotton fabric dyeing, can be in neutral or alkaline bath dyeing dyeing, also can be used for printing of cotton, including direct printing, the printing and dyeing discharge printing. Can also be used for the silk fabric and wool fabric and polyester/cotton blended fabric dyeing.

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	4	5	5	4	4	4-5	5

8.Name: C.I. Solubilised Vat Orange 5 ,C.I.73336

Molecular Structure: Thioindigo class



Molecular Formula: C₂₀H₁₆Na₂O₁₀S₄

Molecular Weight: 590.58

CAS Registry Number: 10126-87-11

Manufacturing Methods : (a) in the presence of Pyridine , with Chlorosulfonic acid treatment C.I. Vat Orange 5 leuco ; (b) used in Pyridine metal (such as copper, iron, zinc, etc) and Sulfur trioxide source (such as Sulfur trioxide, Chlorosulfonic acid or Methyl chlorosulfonate) processing C.I. Vat Orange 5. The above two kinds of circumstances are through with alkali, filter out the insoluble,, remove Pyridine and salting-out.

Properties and Applications: Red light orange. Orange powder. Soluble in water. Meet strong sulfuric acid into purple. Can be used for the cotton fabric dyeing, red light for orange. Can also be used to the cotton fabric dye printing, the white printing, also can be used for silk and wool dyeing. [13]

Standard	Ironing Fastness	Chlorine bleach	Light Fastness	Mercerized	Oxygen bleach	Soaping	
						Fading	Stain
ISO	4	4	4-5	4	4	4	4-5
AATCC	5	4-5	4	4	3		

Properties Of Vat Dye

1. Vat dye is water insoluble and can't be applied directly on textile material.
2. Mainly use for cellulose fibre dyeing but in protein fibre dyeing PH should be controlled.
3. Rubbing fastness is not good.
4. Various shades is found.
5. Dyeing process is difficult.
6. Costly.
7. Washing fastness of vat dye is very good with rating 4-5.

2.3.2 Classification Of Vat Dyes

The vat dyes are to be divided into several groups, depending upon their chemical nature and origin, as follows:

- (a) Indigo, including both natural and Synthetic
- (b) Thio-indigo dyes, containing sulphur.
- (c) Indigo derivatives, such as the brom-indigos; usually not derived directly from indigo itself, but built up synthetically.
- (d) Anthraquinone derivatives, including the various Indanthrene, Marione, Algol dyes, some Helindone, and others.
- (e) Carbazol derivatives, of which Hydron Blue is the chief representative .[14]

2.3.3 Mechanism Of Vat Dyeing Process

Most vat dyes, which require a reducing agent to solubilize them, are less suitable than fiber-reactive dyes for amateurs.

Chemical reactions such as oxidation, reduction, pH control are often necessary; even the dissolution process necessitates measuring out appropriate quantities of caustic soda and sodium hydrosulphite in order to achieve reduction. The dye is soluble only in its reduced (oxygen-free) form. The fiber is immersed repeatedly in this oxygen-free dyebath, then exposed to the air, whereupon the water-soluble reduced form changes color as oxygen turns it to the water-insoluble form.

Indigo is an example of this dye class: it changes from yellow, in the dyebath, to green and then blue as the air hits it.[15]

Stages Of Vat Dyeing

Stages in Dyeing :

- (i) Vatting
- (ii) Dying
- (iii) Oxidation

Theory Of VAT Dyeing:

The simplest arrangement for fabric is to pull the textile material through the dyebath so that the dye can exhaust on to the fabric surface. Low liquor ratios and the addition of common salt or Glauber's salt both promote such exhaustion. In some cases, the addition of acid also promotes exhaustion. If the dye is only partially soluble in water and likely to be exhausted unevenly, the addition of soap or sodium carbonate may promote leveling. A dyeing is considered to be level if all parts of all fibers have been penetrated evenly and completely. Machines for this type of dyeing are called batch machines.

Package and beam machines for yarns.

Jigs for open width fabrics

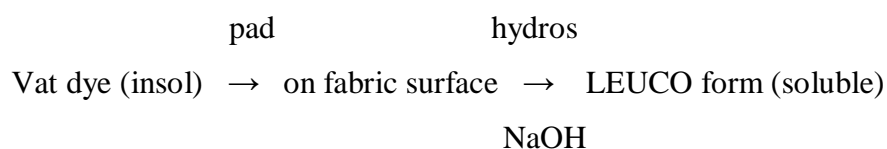
Winches for woven and knitted fabrics in rope form

Jet dyeing machines for knitted fabrics in rope form.

Paddle machines for sewn products like bedspreads

Smith drums for nylon hosiery or special machines for nylon hosiery

If a dye is not soluble in water, as is the case with vats, it may be applied to the fabric as a dispersion by a padder. Once the insoluble vat dye has been uniformly applied to the fabric surface, usually with the aid of special dispersing agents (detergents), it can be solubilized by reaction with a reducing agent, e.g., sodium hydrosulfite ("hydros", $\text{Na}_2\text{S}_2\text{O}_4$) in dilute NaOH. Once it has been converted to its soluble (LEUCO) form, the vat dye can penetrate into the cotton fibers. After adequate time for penetration to occur, the fabric is withdrawn from the bath and oxidized by air or an oxidizing agent such as sodium perborate or hydrogen peroxide. This process is schematically represented below.



Before chemical reducing agents were readily available, vat dyes were converted to their soluble leuco form by fermentation of organic matter in wood tubs called vats. This method of reduction and application is the source of the name for this class of dyes. Once the vat dyes have been regenerated inside the fiber, they are very insoluble. This accounts for their excellent wash fastness. Because they can be applied as a dispersion by padding, solubilized by reduction, and finally re-oxidized when inside the fibers, vats are well-suited to continuous dyeing operations. Such treatments exhibit a number of advantages:

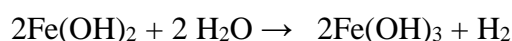
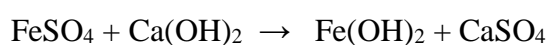
- a) Very efficient use of the dye. All that is made up can be applied.
- b) The insoluble vat is very evenly distributed over the fabric surface, leading to level dyeing
- c) Continuous processes are normally more economical processes than batch processes

[18]

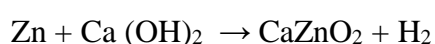
2.3.4 Application Of VatDye:

Most vat dyes are sold in insoluble oxidized form. The first operation therefore, consists of reducing to the leuco compound and dissolving the latter in alkali, a process commonly referred to as vetting. The classical nature vat dyes such as Indigo and Tyrian purple were reduced in fermentation vat.

One of the earlier processes for reducing Indigo, based on the use of chemicals instead of micro – organisms, was with copper's as the colloquial name for ferrous sulphate; the ferrous sulphate was then precipitated which rapidly reacted with the oxygen with the formation of ferric hydroxide, accompanied by the liberation of hydrogen.



Since the Indigo leuco compounds dissolve in mild alkali the excess of calcium hydroxide sufficed for this purpose. the copper's method was followed by the Zinc line vat. Based upon the fact that zinc dissolved in lime water formation calcium zinc ate and hydrogen.



Neither method was entirely satisfactory because the copper's precipitates of either ferric hydroxide or calcium zincates interfered seriously with the dyeing process.[16]

Application Properties:

Light fastness of Vat dyes:

Vat dyes are as a class, the most light fast of all dyes. However, not all individual vat dyes are equally resistant to light. The following are light fastness rating for the unmixed dyes.

Table No. 2.6:Light fastness of vat dyes

S/NO	Color name	Color Index name	Light fastness rating (out of 8)
1	VD01 Yellow	Vat yellow 2	5
2	VD02 Orange	Vat orange 2	5
3	VD03 Red	Vat red 13	7
4	VD04 Blue	Vat blue 6	7 – 8
5	VD08 Violet	Vat violet 1	6

Wash Fastness:

The wash fastness rating of vat dyes is about 4 – 5 .The excellent wash fastness of textile materials, colored with vat dyes is attributed to the large vat dyes molecule as well as its aqueous insolubility. The large vat dye molecule is trapped within the polymer system of the fiber because of its size and aqueous insolubility and it is absorbed within the fiber system by vanderwals forces.[17]

Important Requirements Of Vat Dye Reducing Agent

A level of reducing power (reduction potential) sufficient to reduce all commercial vat dyes to their water soluble form, quickly and economically Conversion of the vat dyes into products from which the original pigment can be restored (no over-reduction).

Various reducing systems for vat dyes have been proposed and used. The most common type of reducing agent used for dyeing with vat dyes is sodium hydrosulphite, commonly known as hydros but more correctly known as sodium dithionite, which has the chemical formula $\text{Na}_2\text{S}_2\text{O}_4$. Although a part of the hydros is used up in the reduction of vat dyes, a large part of it may be destroyed by its reaction with oxygen in the air (oxidation), particularly at higher temperatures.

CHAPTER 03

**SIGNIFICANCE AND SCOPE OF
THE STUDY**

CHAPTER 03

SIGNIFICANCE AND SCOPE OF THE STUDY

3.1 Significance of the Study:

- a) Collection of eight types of knit fabric samples from the industry and pre-treatment of these sample by a suitable recipe.
- b) Dyeing of selected fabric samples with vat dye and then oxidation of dyed samples by physical and chemical oxidation methods.
- c) Testing of colorfastness properties (washing and rubbing) of dyed and oxidized samples according to ISO 105 C₀₃ and ISO 105 X₁₂ and then keeping records of the ratings.
- d) Drawing line diagrams based on colorfastness ratings of eight selected samples and making discussions followed by a conclusion.

3.2 Scope of the Study:

The scope of the present work entails-

Chapter-1 contains the introduction part of the project report.

Chapter-2 presents literature review or theoretical knowledge required for doing this research work.

Chapter-3 includes significance and scope of this project report.

Chapter-4 contains research methodology that includes samples specification, chemicals used, pre-treatment and dyeing procedure, evaluation method etc.

Chapter-5 includes the analysis of collected data after assessing the test samples and line diagrams here to interpret the research results in an organized way.

Chapter-6 finally covers the conclusion part of the report.

CHAPTER 04

RESEARCH METHODOLOGY

CHAPTER 04

RESEARCH METHODOLOGY

4.1 Materials:

For this research we have chosen different types of knitted fabric of 100% cotton different GSM. Sample specification are stipulated in below-

Table 4.1: Sample specification.

Sample No	Sample Name	Fabric Type	Fabric GSM	Yarn Count (Ne)
1	Single Jersey	100% Cotton Knitted	140	28
2	Single Jersey	100% Cotton Knitted	280	30
3	Slub Single Jersey	100% Cotton Knitted	180	28
4	Single Lacoste	100% Cotton Knitted	180	34
5	Fleece	100% Cotton Knitted	280	34
6	(2×2) Rib	100% Cotton Knitted	180	28
7	Interlock	100% Cotton Knitted	220	24
8	Pique	100% Cotton Knitted	220	34

4.2. Methods:

4.2.1.Pretreatment Method:

Recipe for scouring and bleaching:

Detergent= 0.5 g/l (Stock solution 1%)

Caustic soda= 2.0 g/l (Stock solution 1%)

H₂O₂= 5.0 g/l (Stock solution 1%)

Sequestering agent= 0.5 g/l (Stock solution 1%)

Anti-creasing agent= 0.5 g/l (Stock solution 1%)

Anti-foaming agent= 0.3 g/l (Stock solution 1%)

M: L= 1: 30

pH= 10.8

Temperature= 98⁰C

Time= 60 Minute

Material weight= 120 gm.

Calculation:

Total liquor= 120x30=3600 cc

Detergent= (3600x0.5)/ (1000x1%) = 180 cc

Caustic soda= (3600x2)/ (1000x1%) = 720 cc

H₂O₂= (3600x5)/ (1000x1%) = 1800 cc

Sequestering agent= (3600x0.5)/ (1000x1%) = 180 cc

Anti-creasing agent= (3600x0.5)/ (1000x1%) = 180 cc

Anti-foaming agent= (3600x0.5)/ (1000x1%) = 180 cc

Required water= 3600- (180+720+1800+180+180+180) = 360 cc

Process sequence:

- At first we have set the bath at normal temperature.
- We have taken all the chemicals and required water into the bath according to the recipe.
- Then added sample into the bath.
- After that we have raised the temperature up to 98⁰C.
- We have kept the bath at 98⁰C for 60 minutes.
- Finally drained the bath.

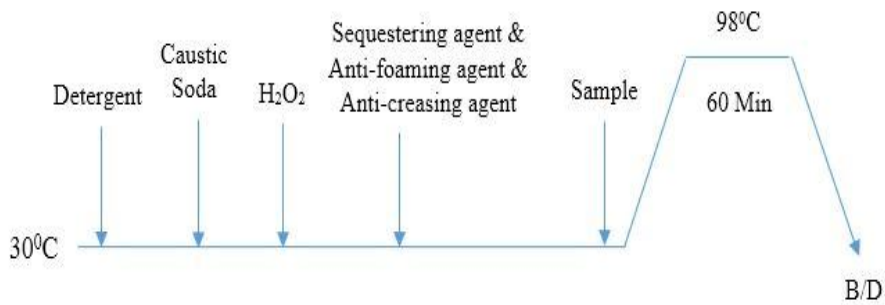


Figure 4.1.: Pretreatment process curve

4.2.2. Dyeing Method:

Recipe of vat dyeing:

Vat dye Blue=1% owf (Stock solution=1%)

NaOH= 1 gm/litre (Stock solution=1%)

Hydrose= 2 gm/litre (Stock solution=1%)

Wetting agent= 0.5 gm/litre (Stock solution=1%)

pH=9.1

Material weight= 15 gm x 8= 120 gm

M: L= 1:30

Temperature= 100⁰ C

Time= 30 min

Calculation:

Vat dye Blue= $(1\% \times 120) / 1\% = 120$ cc

NaOH= $(3600 \times 1 \times 100) / (1000 \times 1) = 360$ cc

Hydrose = $(3600 \times 2 \times 100) / (1000 \times 1) = 720$ cc

Wetting agent= $(3600 \times 0.5 \times 100) / (1000 \times 1) = 180 \text{ cc}$

Total liquor = $120 \times 30 = 3600 \text{ cc}$

Required water = $\{3600 - (120+360+720+180)\} = 2220 \text{ cc}$

Dyeing process:

- At first vat dye and NaOH ,hydrose are taken at required quantity in a bath with required water and have taken on a burner.
- After raising the temperature at 60, we have left the bath on the burner for 20 minutes for vatting.
- Then wetting agent is added on the bath and the samples are also added after wetting.
- We have taken the bath on the burner until the temperature has reached at 100 and then have been waiting for 30 min.
- After 30 minutes later ,dyeing is completed .
- Finally the dyed fabrics are taken out from the dye bath and dried at atmospheric temperature.

Process curve:

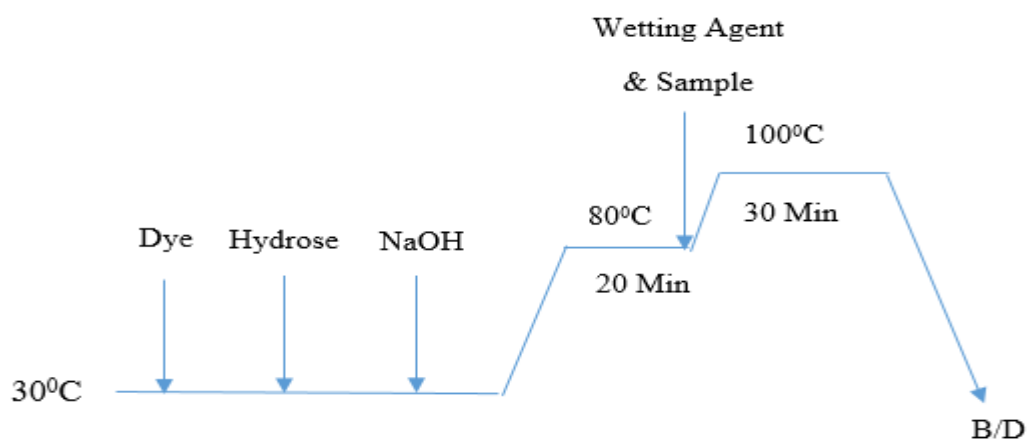


Figure 4.2.: Dyeing process curve

4.2.3 Oxidation Method

Oxidation is mainly carried out at two states. These are..

- Physical oxidation
- Chemical oxidation

Physical oxidation:

After dyeing a cotton fabric or any fabric with vat dye will be subjected to an oxidation treatment by exposure to atmospheric oxygen. This is called “air oxidation” or “physical oxidation”. After physical oxidation the soluble vat dye may convert into insoluble form at the contact of atmospheric air.

Chemical oxidation:

a) Chemical oxidation with H₂O₂

Recipe:

H₂O₂ = 1 gm/litre (Stock solution = 1%)

Material weight = 40 gm

M: L = 1:20

pH = 7

Temperature = 50°C

Time = 10 mins

Calculation:

$H_2O_2 = (1 \times 800 \times 100) / (1000 \times 1\%) = 80 \text{ cc}$

Total liquor = 800 cc

Required water = 800 – 80 = 720 cc

Oxidation process:

- i) At first we have taken required quantity of water, H₂O₂ in a bath.
- ii) Then put it on a burner and 1 minute later we have added the material.

- iii) After reaching the temperature at 50⁰C ,we have waited for 10 minutes.
- iv) After 10 minutes later, we have taken out the samples from the bath and finally dried it at atmosphere.

Process curve:

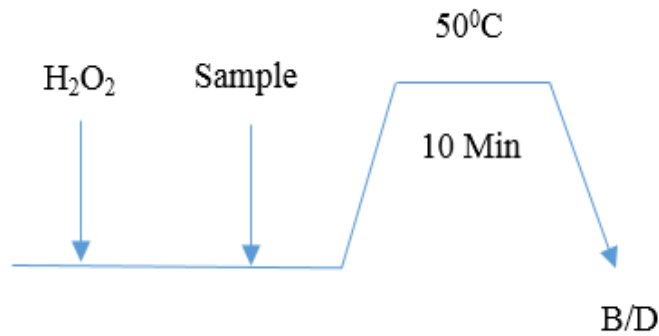


Figure 4.3.:Oxidation process curve for H₂O₂

b) Chemical oxidation with sodium per borate

Recipe:

Sodium per borate = 1 gm/litre (Stock solution = 1%)

Material weight = 40 gm

M: L = 1:20

pH=7.1

Temperature= 50⁰C

Time= 10 mins

Calculation:

Sodium per borate = $(1 \times 800 \times 100) / (1000 \times 1\%) = 80 \text{ cc}$

Total liquor = 800 cc

Required water = $800 - 80 = 720 \text{ cc}$.

Oxidation process:

- i) At first we have taken required quantity of water ,sodium perborate a bath.
- ii) Then put it on a burner and 1 minute later we have added the material.
- iii) After reaching the temperature at 50⁰C ,we have waited for 10 minutes.
- iv) After 10 minutes later ,we have taken out the samples from the bath and finally
- v) dried it at atmosphere.

Process curve:

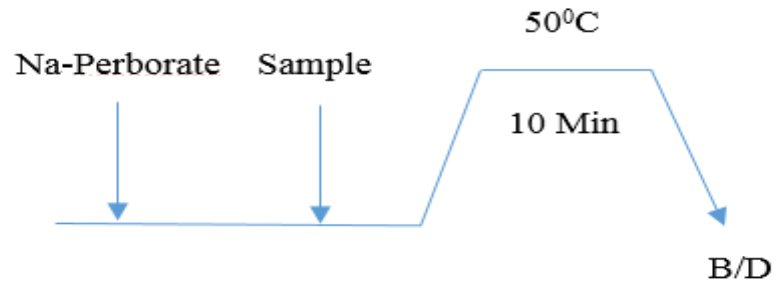
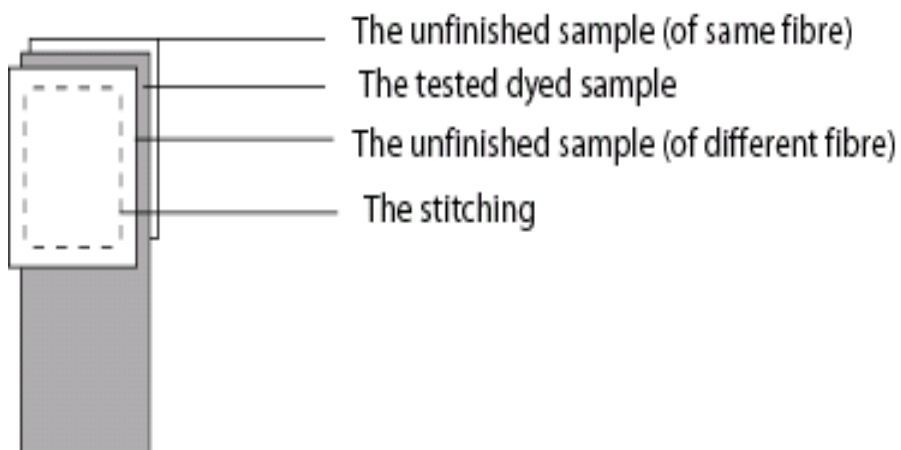


Figure 4.4.:Oxidation process curve for sodium perborate

4.2.4. Testing Method

a) Color fastness to wash:

Specimen preparation:

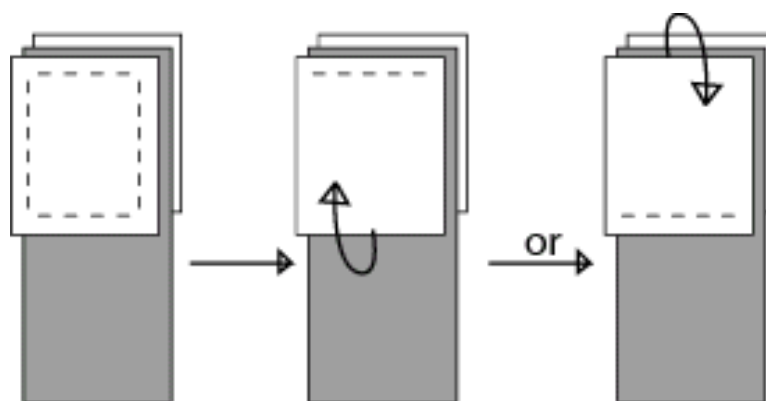


The tested sample is prepared as below:

- ❖ A $10 \times 4 \text{ cm}^2$ dyed sample fabric to be tested is taken.
- ❖ Another two pieces of fabric sample of $5 \times 4 \text{ cm}^2$ which are scoured, bleached but undyed is also taken.
- ❖ Now the dyed sample is placed at one side of the undyed samples covering $5 \times 4 \text{ cm}^2$ area and stitched at the four edges, leaving $5 \times 4 \text{ cm}^2$ exposed, thus the specimen is ready for test.

Washing process:

- According to ISO iii method 5gm/litre ISO reference detergent need to be taken .
- As the M:L is 1:50
Sample weight is 20 gm
So total liqure(20×50)= 1000cc
Detergent= $(1000 \times 5 / 1000)\text{gm} = 5\text{gm}$
- Then we have added 5gm detergent into 400cc water.
- After that we have put the sample into the solution taking the bath onto the burner.
- When temperature has reached at 60^0c we have taken the bath for 30 minutes on the burner.
- After 30 mins later we have taken samples out from the bath and rainsed with cold water .
- Finally the stitch is removed as following.



Assessment Technique:

The exposed part is compared with original dyed fabric with the help of grey scale.

b)Color fastness to rubbing:

Rubbing fastness is the resistance to fading of dyed textiles when rubbed against a rough surface.

- ❖ The fastness to crocking or rubbing is widely used on a variety of fabric to evaluate the transfer of surface dye from the test fabric when it is applied surface friction or rubbed against a rough surface.
- ❖ Two types of rubbing test are done-
 - ✓ Dry rub
 - ✓ Wet rub

Apparatus:

- ❖ Crock meter
- ❖ Grey scale for staining

Procedure
























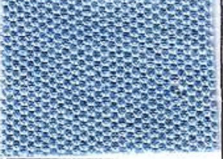
- Test specimen 15cm x is placed on the base of the Crock meter.
- A square of white test cloth (5cm x 5cm) which is of plain weave, desized, bleached but without finished cotton fabric is taken.
- White test cloth is attached to the finger of the crock meter.
- This finger is used in rubbing action on the sample specimen
- Rubbing is done to and fro, 10 cycles at 10seconds, i.e.20 rubs in 10s and finger pressure on the specimen is 9N.
- Rubbing test is done both for warp way and weft way.
- For dry and wet rubbing test, separate sample is used.
- For wet rubbing, the sample is dry but crocking cloth is wet. For wetting, M:L ratio is maintained not less than 1:50,water is drained after wetting and not squeezed.

Assessment Technique:

Generally degree of staining in the white cloth is determined by grey scale comparing with the dyed fabric.

SAMPLE ATTACHMENT





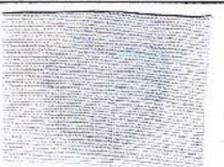



















A. Attachment of Samples Dyed with Vat Dye

Sample No.	Attached Samples (Physical Oxidation)	Sample No.	Attached Samples (Chemical Oxidation by H_2O_2)	Sample No.	Attached Samples (Chemical Oxidation by $NaBO_3$)
1		9		17	
2		10		18	
3		11		19	
4		12		20	
5		13		21	
6		14		22	
7		15		23	
8		16		24	

























B. Attachment of Colorfastness to Rubbing (Dry) Test Samples

Sample No.	Attached Samples (Physical Oxidation)	Sample No.	Attached Samples (Chemical Oxidation by H_2O_2)	Sample No.	Attached Samples (Chemical Oxidation by $NaBO_3$)
1		9		17	
2		10		18	
3		11		19	
4		12		20	
5		13		21	
6		14		22	
7		15		23	
8		16		24	

C. Attachment of Colorfastness to Rubbing (Wet) Test Samples

Sample No.	Attached Samples (Physical Oxidation)	Sample No.	Attached Samples (Chemical Oxidation by H_2O_2)	Sample No.	Attached Samples (Chemical Oxidation by $NaBO_3$)
1		9		17	
2		10		18	
3		11		19	
4		12		20	
5		13		21	
6		14		22	
7		15		23	
8		16		24	

D. Attachment of Colorfastness to Wash Test Samples

Sample No.	Attached Samples (Physical Oxidation)	Sample No.	Attached Samples (Chemical Oxidation by H_2O_2)	Sample No.	Attached Samples (Chemical Oxidation by $NaBO_3$)
1		9		17	
2		10		18	
3		11		19	
4		12		20	
5		13		21	
6		14		22	
7		15		23	
8		16		24	

CHAPTER 05

DATA ANALYSIS & FINIDINGS

CHAPTER 05

DATA ANALYSIS & FINIDINGS

5.1 Data analysis and discussion for colorfastness to washing of different samples:

After testing colorfastness to washing of different samples the ratings were recorded in the **Appendix-A**. The colorfastness ratings have been used here to draw the following **Figure 5.1**.

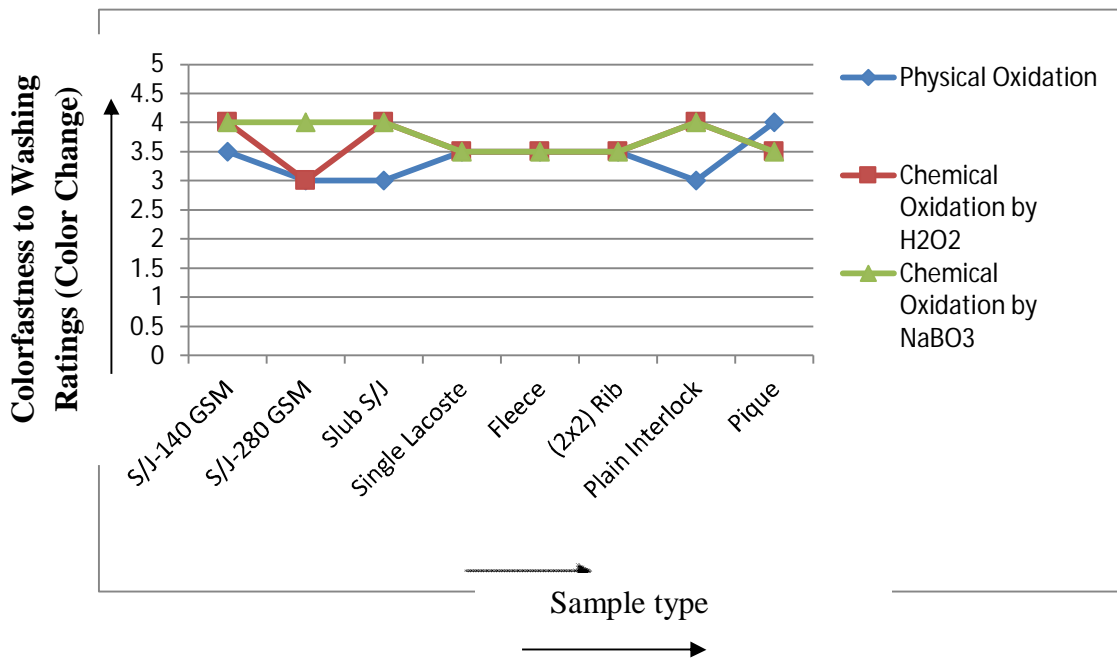


Figure 5.1: Line diagram represents the colorfastness to washing of eight cotton samples under three conditions.

The diagram has been drawn for colorfastness to dry rubbing is a line diagram. In this line diagram, sample type is indicated horizontally or along the X axis and colorfastness rating is indicated vertically or along the Y axis.

From the line diagram we can see that for physical oxidation colorfastness rate is below 5. S/J-280 GSM, Plain interlock, Slub S/J samples are showing the same rating of 3. On the otherhand S/J-140 GSM, Fleece, 2×2 Rib, Single lacost are showing the same rating of 3-4 .

In case of chemical oxidation by H_2O_2 , S/J-140 GSM, Slub S/J, Plain interlock are showing the same result with the rating of 4. 3-4 rate of color fastness is shown by Single lacost, Fleece, 2×2 Rib, Pique. S/J-280 is showing the color fastness rate of 3.

In case of chemical oxidation by $NaBO_3$, S/J-140 GSM, S/J-280 GSM, Slub S/J, Plain interlock and Single lacost, Fleece, 2×2 Rib are showing the result with the rateing accordingly of 4 and 3-4 . 4-5 rate of colorfastness is only shown by Pique.

Finally we can say that chemical oxidation by $NaBO_3$ for Pique is showing the best result with colorfastness rating of 4-5 .

5.2 Data analysis and discussion for colorfastness to dry rubbing of different samples:

After testing colorfastness to dry rubbing of different samples the ratings were recorded in the **Appendix-B**. The colorfastness ratings have been used here to draw the following **Figure 5.2**.

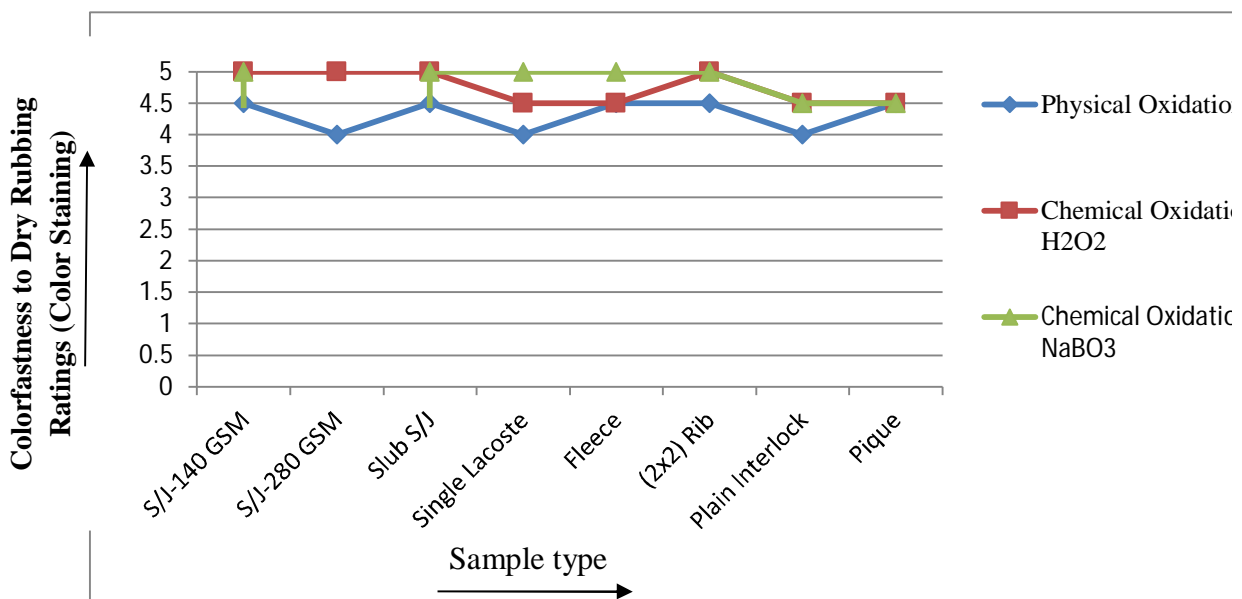


Figure 5.2: Line diagram represents the colorfastness to dry rubbing of eight cotton samples under three conditions.

The diagram has been drawn for colorfastness to dry rubbing is a line diagram. In this line diagram, sample type is indicated horizontally or along the X axis and colorfastness rating is indicated vertically or along the Y axis.

From the line diagram we can see that for physical oxidation colorfastness rate is below 5. S/J-280 GSM, Single lacost, Plain interlock samples are showing the same rating of 4. On the other hand S/J-140 GSM, Slub S/J, Fleece, 2×2 Rib, Pique are showing the same rating of 4-5.

In case of chemical oxidation by H₂O₂, S/J-140 GSM, S/J-280 GSM, Slub S/J, 2×2 Rib are showing the same result with the rating of 5. 4-5 rate of color fastness is shown by rest samples.

In case of chemical oxidation by NaBO₃, all the samples excluding plain Interlock and Pique are showing the result of highest rating of 5.

Finally we can say that chemical oxidation by NaBO₃ for maximum samples showing the best result with colorfastness rating of 5.

5.3 Data analysis and discussion for colorfastness to wet rubbing of different samples:

After testing colorfastness to wet rubbing of different samples the ratings were recorded in the **Appendix-B**. The colorfastness ratings have been used here to draw the following **Figure 5.3**.

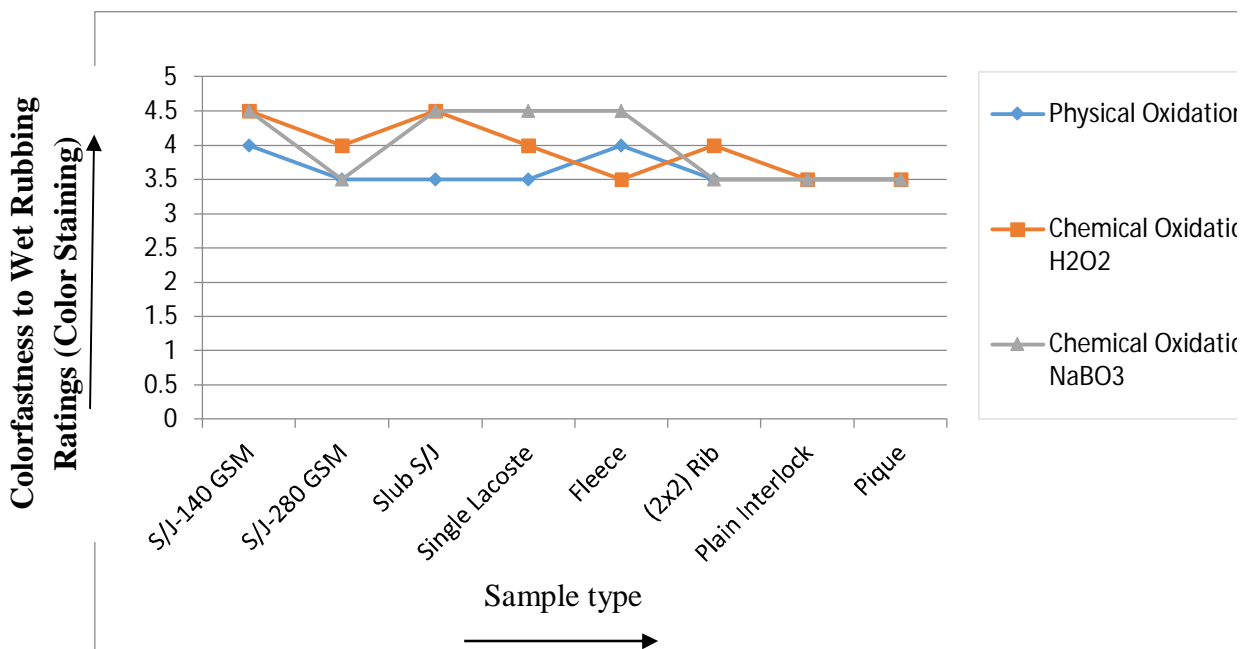


Figure 5.3: Line diagram represents the colorfastness to wet rubbing of eight cotton samples under three conditions.

The diagram has been drawn for colorfastness to wet rubbing is a line diagram. In this line diagram, sample type is indicated horizontally or along the X axis and colorfastness rating is indicated vertically or along the Y axis.

From the line diagram we can see that for physical oxidation, colorfastness rate is equal or below 4. S/J-280 GSM, Single lacost, Slub S/J, 2×2 Rib, Plain interlock, Pique samples are showing the same rating of 3-4. On the otherhand S/J-140 GSM and Fleece are showing the same rating of 4.

In case of chemical oxidation by H_2O_2 , S/J-140 GSM, Slub S/J are showing the same result with the rating of 4-5. 4 and 3-4 rate of color fastness is shown accordingly by S/J-280 GSM, Single lacost, 2×2 Rib and Fleece, Plain Interlock, Pique.

In case of chemical oxidation by $NaBO_3$, S/J-140, Slub S/J, Single lacost, Fleece and S/J-280, 2×2 Rib, Plain interlock, Pique are showing the fastness rate accordinly of 4-5 and 3-4 .

Finally we can say that same amount of samples of chemical oxidized by $NaBO_3$ are showing colorfastness rating of 4-5 and 3-4 .

CHAPTER 06
CONCLUSION

CHAPTER 06

CONCLUSION

In this project we have studied how different oxidizing agents effect on various types dyeing cotton fabric with vat dye including different test like rubbing, wash fastness test .The study was conducted with lot of limitations.With the limitations of lab facilities we have finished the project well ahead.Also the project work basically helped us to meet the knowledge about different fabric ,dyeing process with vat dye, process of oxidation with different effect on different fabric and so on.In this study we have used eight types of cotton fabric,if we had used more types of fabric during research we will get more knowledge with their result analyzing them.Here we have used only two types oxidizing agent, more types of oxidizing agents will lead us to gain more knowledge with their effect on different types cotton fabric.

So finally completing the whole research on “the effect of oxidizing agents on dyeing cotton fabric with vat dye”,we have reached at the end with the result.

- In case of colorfastness to wash we have found only pique sample chemically oxidized by NaBO_3 with good color rating of 4-5.
- For colorfastness to dry rubbing ,we have found maximum samples chemically oxidized by NaBO_3 showing the highest colorfastness rating of 5 than other oxidized samples.
- And only maximum samples chemically oxidized by NaBO_3 are showing the best rating of 4-5 than other oxidized samples in case of colorfastness to dry rubbing.

So, finally we can say that after completing the research we have found the end of the project with the result of S/J-140 GSM, S/J-280 GSM, Slub S/J ,Single lacost,Fleece,2×2 Rib sampes chemically oxidized by NaBO_3 have the highest colorfastness rate of 5.

APPENDIX-A

(Colorfastness to Washing Ratings of Different Samples)

Table A.1: Colorfastness to washing ratings of different cotton dyed samples.

Sample No.	Sample Name	Physical Oxidation	Chemical Oxidation by H ₂ O ₂	Chemical Oxidation by Sodium Per borate
		Colorfastness to Washing Ratings (Color Change)		
1.	Single Jersey-140 GSM	3-4	4	4
2.	Single Jersey-280 GSM	3	3	4
3.	Slub Single Jersey	3	4	4
4.	Single Lacoste	3-4	3-4	3-4
5.	Fleece	3-4	3-4	3-4
6.	(2×2) Rib	3-4	3-4	3-4
7.	Plain Interlock	3	4	4
8.	Pique	4	3-4	3-4

APPENDIX-B

(Colorfastness to Rubbing Ratings of Different Samples)

Table B.1: Colorfastness to rubbing ratings of different cotton dyed samples.

Sample No.	Sample Name	Physical Oxidation	Chemical Oxidation by H ₂ O ₂		Chemical Oxidation by NaBO ₃		
		Colorfastness to Rubbing Ratings (Staining)					
		Dry	Wet	Dry	Wet	Dry	Wet
1.	Single Jersey-140 GSM	4-5	4	5	4-5	5	4-5
2.	Single Jersey-280 GSM	4	3-4	5	4	4-5	3-4
3.	Slub Single Jersey	4-5	3-4	5	4-5	5	4-5
4.	Single Lacoste	4	3-4	4-5	4	5	4-5
5.	Fleece	4-5	4	4-5	3-4	5	4-5
6.	(2×2) Rib	4-5	3-4	5	4	5	3-4
7.	Plain Interlock	4	3-4	4-5	3-4	4-5	3-4
8.	Pique	4	3-4	4-5	3-4	4-5	3-4

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