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EFFECT OF ALKALI TREATMENT & DYEING ON FIBRILLATION PROPERTIES OF LYOCELL FIBER

A PROJECT REPORT

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BONAFIDE CERTIFICATE

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ABSTRACT

Lyocell is a new generic name given to a cellulosic fiber which is produced under an environmentally friendly process by dissolving cellulose in the tertiary amine oxide N-methylmorpholine-N-oxide (NMMO). Lyocell fiber shows some key advantageous characteristics over other cellulosic fibers; for instance, a *high dry and wet tenacity and high wet modulus*.

However, the fiber also shows an extensive tendency to fibrillate in the wet state, which causes the formation of longer and more oriented crystalline regions and smaller but more oriented amorphous regions in the fiber structure. This fibrillar structure is responsible for the high fiber tenacity but low lateral cohesion, especially when subjected to mechanical stress in the swelled state. Even though formation fibrillation characteristic has been shown in *advantageous in few applications* such as peach *skin* appearance of the surface is required. But it has been proven to be *disadvantageous for some other applications*, such as the launderability of the product and difficulty to control the uniformity of color uptake during dyeing, Pills formation and streak marks in dyeing. In textile processing, alkali treatment is an important stage. This work is a report on a study of the fibrillation tendency in different types of alkali-treated and dyeing with poly functional reactive dyes of lyocell fibers. Basically Lyocell dyed with some poly-functional reactive dyes exhibited excellent resistance to the fibrillation and good dye uptake, whereas mono-functional and di- functional and bi-functional reactive dyes were not effective to depress the fibrillation.

In the present study, report on a fibrillation tendency of lyocell, in different types of alkali-treating, and dyeing of poly functional reactive dyes. The effect of alkali concentration, poly functional reactive dyes on fibrillation tendency can be absorbed with the help of Pilling Resistance, Abrasion Resistance and SEM Analysis; also various physical and chemical properties also Studied.

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LIST OF ABBREVIATIONS

AATCC	American Association of Textile Chemists and Colourists
ASTM	American Standards of Testing Materials
CSP	Count Strength Product
DMF	Di –Methyl Formamide
DMAC	Di-Methyl Acetamide
DMSO	Di- Methyl Sulfoxide
DP	Degree of Polymerization
HMPA	Hexa Methyl-Phosphoric Triamide
GSM	Grams per Square meter
H_2O_2	Hydrogen Peroxide
K OH	Potassium Hydroxide
Kpa	Kilo pascal
K/S	Colour strength value
Li OH	Lithium Hydroxide
Na OH	Sodium Hydroxide
NDMA	Nitroso Di-Methyl Amine
N_e	English count
NMMO	N- Methyl Morpholine-N- oxide
PF	Poly Functional
SEM	Scanning Electron Microscope
STEM	Scanning Transmission Electron Microscopes
TEM	Transmission Electron Microscopy
Tex	Universal Count
TMAH	Tetra Methyl- Ammonium Hydroxide

CHAPTER 1
INTRODUCTION

Lyocell is the *first in a new generation of cellulosic fibers*. The development of lyocell was driven by the desire for a cellulosic fiber which exhibited an improved cost and performance profile compared to other cellulosic fibers (Nechwatal A et al 1996). The other main driving force was the continuing demands for industrial processes to become more environmentally responsible and utilize renewable resources as their raw materials. The resultants lyocell fiber meets both demands. The first commercial samples were produced in 1984 and fiber production has been increasing rapidly ever since. This is derived from wood pulp produced from sustainable managed forests (Lenz et.al 1992).

Lyocell has all the benefits of being a cellulosic fiber, in that it is fully biodegradable, it is an absorbent and excellent in handle and etc. It has a relatively high strength in both the wet and dry state which allows for the production of finer yarns and lighter fabrics (Fink et.al 2001). Fabrics produced from lyocell fibers are breathable, moisture absorbent and have excellent dimensional stability (Okubayashi S Griesser U 2004)The high strength also facilitates its use in various mechanical and chemical finishing treatments both under conventional and extreme conditions. The physical characteristics of lyocell also result in its excellent blending characteristics with all type of fibers namely linen, cashmere, silk, and wool (Braunesis F & Eibl M 1998).

In common with other highly oriented cellulosic fibers such as cotton, cuprammonium and polynosic rayon, lyocell fibrillates but its ease of fibrillation is greater. In the swollen state lyocell has an extensive fibrillation tendency owing to linear high crystalline fibrillar morphology (Rohrer C et.al 2001). The fibrillation tendency of lyocell enables this fiber to be used in specific finishing effects such as *peach skin, silk touch and soft denim*. On the other hand, the fibrillations induce e.g. *rope marking defect in hank finishing, graying of dyed fabrics and a change of handle of clothes* that spoils garments features. Efforts to control the fibrillation tendency in lyocell fibers include dyeing with reactive dyestuffs and treating fabrics with crosslinking agents. Some of the most important steps in fiber processing involve alkalis, such sodium hydroxide or sodium carbonate, and some fiber response to alkali treatments is an important criterion (Nemec H 1994).

Fibrillation may lead to pilling and therefore spoil fabric appearance and touch. Pill formation is a common problem mainly in knitted fabrics made not only from synthetic fibers but also from natural fibers, man-made cellulosic's and their blends because no consumers accept the undesirably pilled garments (Nemec H 1994).

1. 1 OBJECTIVE OF THE PROJECT

To control degree of fibrillation formation with the help of

- *Treating with Various Alkali Treatments and followed by Dyeing with Poly Functional Reactive Dyes*
- Analyzing of Physical and chemical properties of treated lyocell
- Determining the Suitable treatment for arresting the fibrillation formation.

In the present work an attempt had made to reduce the fibrillation formation in the lyocell fiber.

CHAPTER 2

LITERATURE REVIEW

2.1 LYOCELL

Lyocell is a regenerated cellulose fiber made from dissolving pulp (bleached wood pulp). It was first manufactured in 1987 by Courtaulds Fibers UK. As of 2010 it is manufactured by Lenzing AG of Lenzing, Austria, under the brand name "Lyocell by Lenzing", and under the brand name Tencel by the Tencel group, now owned by Lenzing AG. Lyocell as "a cellulose fabric that is obtained by an organic solvent spinning process". It classifies the fiber as a sub-category of rayon (Jakob B and Agster E 1998).

Lyocell was introduced to consumers in 1991 and originally marketed as a type of Rayon. Lyocell shares many properties with other cellulosic fibers such as cotton, linen, ramie and rayon. Some main characteristics of Lyocell fibers are that it is soft, absorbent, and very strong when wet or dry, and resistant to wrinkles; it can be machine- or hand-washed or dry cleaned, it drapes well, and it can be dyed many colors, as well as simulating a variety of textures like suede, leather, or silk (Chavan RB and Patra AK 2004). The fiber is used to make textiles for clothing and other purposes.

Lyocell fiber has a high degree of orientation and crystallinity and higher molecular weight than viscose. As a result, fiber strength and modules of fiber are higher than those for regenerated cellulosic fibers as well as polyester staple fibers. Like cotton, Lyocell fabric results in outstanding wear comfort (Chavan RB and Patra AK 2004).

Fabrics produced from Lyocell are breathable and moisture absorbent and have high dimensional stability. Lyocell fabric distinguishes them by the specific property to fibrillate in wet state under impact of external mechanical effects. Fibrillation characteristics of Lyocell make possible the creation of entirely new handle.

Due to special physical properties of the fiber during processing, a wide variety with novel effects like peach skin, silk touch and soft denim can be achieved for high grade fashion garments. Lyocell fibers blend well with other natural or synthetic fibers. It is stronger than cotton. It has the breathability and absorbency of a natural fiber, the durability, and easy care performance of a man-made one. Due to its softness it can blend easily with other fibers. Lyocell fabric blends are widely used in apparel and other fashion garments (Colom X and Carrillo F 2002).

2.2 LYOCELL FIBER MANUFACTURING

2.2.1 Production method

Lyocell is created through a process called solvent spinning. The wood pulp is dissolved in N-Methylmorpholine N-oxide, creating a solution called ***"dope"*** (Jakob B and Agster E 1998). The dope is then pushed through a spinneret to form the individual fibers. After the dope has been spun into Lyocell fibers, it is washed and the chemicals are retrieved from the water, purified, and recycled. Since there is little by product, this process is labeled as relatively eco-friendly. However, it uses a substantial amount of energy and the solvent is a by-product of petrol production.

2.2.2 Fiber Manufacturing

The starting material for Lyocell and viscose are the same, i.e. wood pulp. No cellulose derivative is formed in the former, while viscose rayon manufacturing involves formation of intermediate derivative. Lyocell is manufactured by a direct dissolving process using an organic cyclic polar solvent, namely N- methyl morpholine-N- oxide (NMMO) (Nemec H 1994). This solvent is non- toxic and easily regenerated. NMMO has higher cellulose dissolving capacity than the other organic polar solvents, like DMSO, DMF, DMAC, NDMA, HMPA, etc.

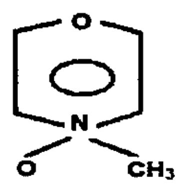


Figure 2.1 Chemical Structure of NMMO

The NMMO solution used is a 50:50 (w/w) mixture of solvent is AM and the common name is Amine oxide. The melting point of the monohydrate solvent is about 76°C. NMMO can be produced from N-methyl morpholine and hydrogen peroxide as per the following reaction:

$$O\,(C_4H_6)\,NCH3 \xrightarrow[CO_2]{H_2O_2} O\,(C_4H_6)\,NOCH3 + H_2O$$

N-Methyl Morpholine *N- Methyl Morpholine – N- Oxide*

Wood pulp is dispersed in concentrated aqueous NMMO and dissolved under exertion of intensive shear forces and simultaneous evaporation of water. The pulp used is an industrial dissolving pulp, having a DP of 750, with 96% cellulose. The starting point of the process is a suspension of approximately 13% cellulose, 20% water and 67% NMMO. Dissolution of cellulose in NMMO is done at 120° (Temperature more than 125-130°C being unsafe for NMMO), resulting in a highly viscous solution (http://www.lenzing.com/lyocell/production.20954.html)

. The solution is filtered and then extruded into a water bath through fine jets. As the solvent is washed out, the fibers formed into fine filaments are collected as tow, from which the staple fiber is produced. The surplus water is evaporated off and the remaining concentrated NMMO is recycled into the process (Nechwatal A., Nicolai M. and Mieck K.- P. 1996).

2.3 MANUFACTURING PROCESS

2.3.1 Lyocell Process Sequence

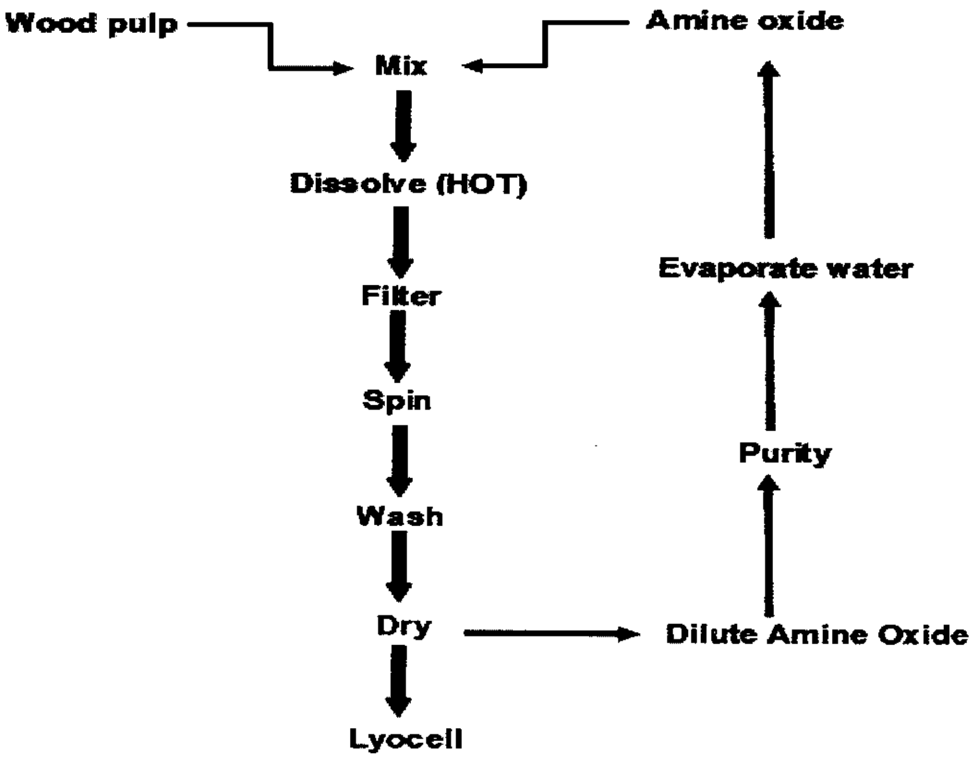


Figure 2.2. Process sequence for Lyocell Manufacturing
(http://www.lenzing.com/lyocell/production.20954.html)

2.3.2 Preparing the wood pulp

The hardwood trees grown for Lyocell production are harvested by loggers and trucked to the mill. At the mill, the trees are cut to 20 ft (6.1m) lengths and debarked by high-pressure jets of water. Next, the logs are fed into a chipper, a machine that chops them into squares little bigger than postage stamps.

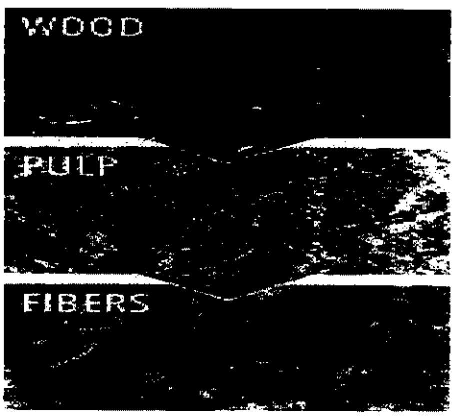


Figure 2.3 Lyocell process from wood

Mill workers load the chips into a vat of chemical digesters that soften them into a wet pulp. This pulp is washed with water, and may be bleached. Then, it is dried in a huge sheet, and mill workers roll it onto spools (http://www.lenzing.com/lyocell/processparameters.543.pdf). The sheet of cellulose has the consistency of thick poster board paper. The roll of cellulose is enormous, weighing some 500 lb (227 kg).

2.3.3 Dissolving the cellulose

At the Lyocell mill, workers unroll several spools of cellulose and break them into one inch squares. The workers then load these squares into a heated, pressurized vessel filled with amine oxide.

2.3.4 Filtering

After a short time soaking in the solvent, the cellulose dissolves into a clear solution. It is pumped out through a filter, to insure that all the chips are dissolved.

2.3.5 Spinning

Next, the solution is pumped through spinnerets. These are devices used with a variety of manmade fibers. Something like a showerhead, the spinneret is pierced with small holes, and when the cellulose is forced through it, long strands of fiber come out. The fibers are then immersed in another solution of amine oxide, diluted this time. This sets the fiber strands. Then, they are washed with de-mineralized water (J. M. Taylor and A. L. Harnden 1997).

2.3.6 Drying and finishing

The Lyocell fiber next passes to a drying area, where the water is evaporated from it. The strands at this point pass to a finishing area, where a lubricant is applied. This may be soap or silicone or other agent, depending on the future use of the fiber. This step is basically a detangled, making the future steps of carding and spinning into yarn easier.

2.3.7 Final steps

The dried, finished fibers are at this stage in a form called tow. Tow is a large untwisted bundle of continuous length filaments. The bundles of tow are taken to a crimper, a machine which compresses the fiber, giving it texture and bulk. The crimped fiber is carded by mechanical carders, which perform an action like combing, to separate and order the strands. The carded strands are cut and baled for shipment to a fabric mill (Chavan RB and Patra AK 2004). The entire manufacturing process, from unrolling the raw cellulose to baling the fiber, takes only about two hours. After this, the Lyocell may be processed in a wide assortment of ways. It may be spun with another fiber, such as cotton or wool. The yarn can be woven or knit like any other fabric, and given a variety of finishes, from soft and suede-like to silky.

2.3.8 Recovery of the solvent

The amine oxide used to dissolve the cellulose and set the fiber after spinning is recovered and re-used in the manufacturing process. The dilute solution is evaporated, removing the water, and the amine oxide is routed for re-use in the pressurized vessel in step 2. Ninety-nine percent of the amine oxide is recoverable in the typical Lyocell manufacturing process.

2.4 PROPERTIES AND CHARACTERISTICS

Lyocell is a manufactured fiber, but it is not synthetic. It is made from wood pulp harvested from tree farms for this purpose. Because it is made from a plant material, it is cellulosic and possesses many properties of other cellulose fibers, such as cotton, linen, ramie, and rayon - another manufactured but non-synthetic fiber. In many ways, Lyocell is more similar to cotton than it is to rayon. Like other cellulosic fibers, it is breathable, absorbent, and generally comfortable to wear.

In fact, Lyocell is more absorbent than cotton and silk, but less so than wool, linen, and rayon. It can take high ironing temperatures, but like other cellulosic's will scorch, not melt, if burned, and is susceptible to mildew and damage by silverfish. Cellulosic fibers are not resilient, which means they wrinkle.

Lyocell has moderate resiliency. It does not wrinkle as badly as rayon, cotton, or linen, and some wrinkles will fall out if the garment is hung in a warm moist area, such as a bathroom after a hot shower. A light pressing will renew the appearance, if needed. Also, slight shrinkage is typical in Lyocell garments. Stability, overall, is similar to that of silk and better than cotton or linen. Lyocell has strength and durability. It is the strongest cellulosic fiber when dry, even stronger than cotton or linen and is stronger than cotton when wet (Chavan RB and Patra AK 2004).

Lyocell is much stronger than rayon when wet. This property of high wet strength usually determines the extent to which fabrics can be machine washed successfully. Other desirable properties of Lyocell are its luster and soft drape which makes it an aesthetically pleasing fiber. Since it is a manufactured fiber, the diameter and length of fibers can be varied.

Lyocell can be made into microfibers (very fine fibers), offering depth and body to fabrics combined with luxurious drape. Short staple length fibers give a cotton-like look to fabrics. Long filament fibers are successful in silk-like end uses. Lyocell blends well with other fibers including wool, silk, rayon, cotton, linen, nylon, and polyester (Nicolai M et.al 1998). It successfully takes many finishes, both functional and those designed to achieve different surface effects and dyes easily. Overall, Lyocell is a versatile fiber with many desirable properties.

The Properties of Lyocell (Chavan RB and Patra AK 2004):

- Stronger than any other cellulosic fibers, especially when wet
- Easy to process into yarns and fabrics alone or in blends
- Easy to blend (unique fiber presentation)
- Easy to spin to fine count yarns
- Very stable in washing and drying and Thermally stable
- Easy to dye to deep vibrant colors
- Capable of taking the latest finishing techniques to give unique drape
- Comfortable to wear

Table 2.1. Physical Properties of Lyocell and Some Other Fibers.

Property		Lyocell	Cotton	Viscose	Modal	Polyester
Count (dtex)		1.4-1.7	1.5-1.8	1.7	1.7	1.7
Tensile strength, cN/tex	Dry	38-42	20-34	22-26	34-36	55-60
	Wet	34-38	25-30	10-15	19-24	54-58
Elongation, %	Dry	14-16	7-11	17-25	13-15	25-30
	Wet	16-18	11-14	21-30	13-15	25-30
Moisture uptake,%		11.5	8	13	12.5	0.4
Water- retention capacity,%		60-70	45-55	90-100	75-80	3-4
Initial wet modulus, 5%		250-270	100-200	40-60	100-120	210
Loop strength, cN/tex		19	20-26	6	8	11
DP value		500-600	2300-3000	250-350	300-600	--

2.5. SPECIALTY PROPERTIES

2.5.1 Water, most vital for life, is managed uniquely by Lyocell properties

Lyocell absorbs excess liquid and quickly releases it again into the atmosphere. The true nature of Lyocell can be found in this perfect interaction between and clothing system (Goswami P et. al 2007).

Lyocell

Polyester

Figure 2.4 Lyocell absorbs full water content

2.5.2 Moisture (Vapor) Management with extreme air humidity

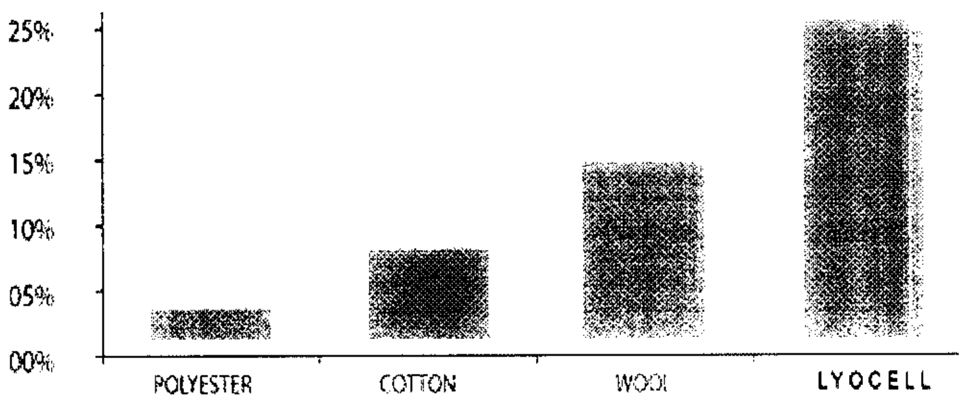


Figure 2.5 Moisture management of Lyocell

The graph shows the increase of moisture in a textile when the textile is moved from a relative humidity of 65% to a relative humidity of 100% (Nicolai M et. al 1998). The moisture refers to the weight of the dry textile.

2.5.3 Lyocell for sensual smoothness

Comparison of the fiber surfaces makes the difference patently obvious. Lyocell fiber has a smoother and more supple surface than wool or cotton. Wool tends to have a scaly surface, while cotton is irregular and rough (Yi-Jun Pan, Chien-Kuo Yen 2005).

Cotton ***Wool*** ***Lyocell***

Figure 2.6 Various fibers surface

2.5.4 Bio – degradable fiber

Lenzing fibers are made from the sustainable, renewable resource wood and combine their natural properties with the advantages of a man-made fiber. They are fully biodegradable and provide natural absorbency and softness to nonwoven products (R. S. Blackburn 2005).

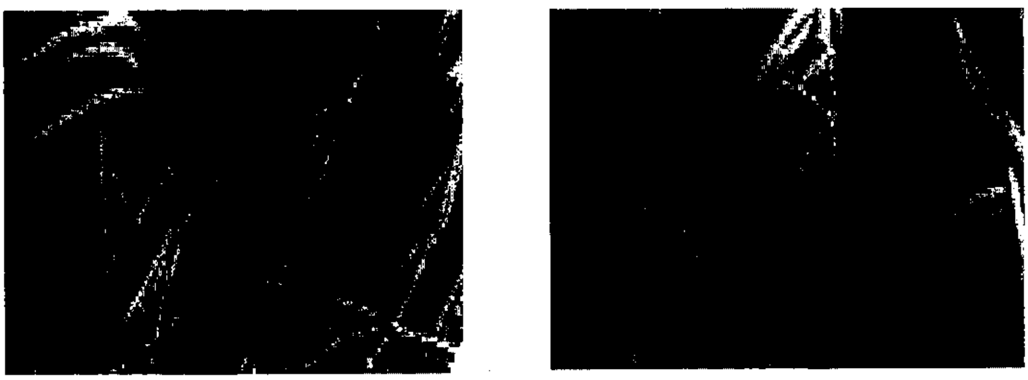
0 Day Soil Burial ***4 Days Soil Burial*** ***7 Days Soil Burial***

Figure 2.7 Bio-degradation properties of lyocell

2.6 FIBRILLATION

It is one of the important properties of Lyocell. Fibrillation is the longitudinal splitting of a single wet fiber into microfibers of <1-4 μ in diameter (Nemec H. 1994), caused by mechanical stress, fibrillation normally takes lack of lateral cohesion. When wet, swelling of the porous regions of the fiber breaks the hydrogen bond linking the crystalline units and forces them apart (Rohrer C. Retzl P. and Firgo H. 2001). When this structure is subjected to mechanical action, the outer crystalline region can break and peel away from the main fiber but remains attached like banana peel (Okubayashi S. and Bechtold T. 2004).

The fibrils formed can be so fine that they become virtually transparent and give a frosty appearance to the finished fabric (Yi-Jun Pan, Chien-Kuo Yen 2005). The *samples figure 2.8 shows* an example of a non-fibrillated (a) and a fibrillated (b) lyocell fiber. The fibrillated fabric gives frosty appearances. In case of extreme fibrillation, the loose fibers on the surface of the fabric fibrillate and then tangle together to form very light colored pills. The appearance of the fabric becomes totally unacceptable.



(a) (b)

Figure 2.8 Example of a non-fibrillated (a) and a fibrillated (b) lyocell fiber.

If fibrillation is not controlled, the microfibers become entangled, giving serious problems of 'pilled' appearance. It also weakens the mother fiber. The fibrillation effect can be advantageous for creating fabrics with an attractive appearance and appealing hand called 'peach-skin effect' (M. Nicolai., Nechwatal A 2006), but for some other applications, it is desirable to eliminate the fibrils.

There are two forms of fibrillation – primary and secondary. The first one consists of long and irregular fibrils which can get entangled, leading to an extremely matted appearance. The secondary form, produced deliberately, is responsible for the fabric's attributes. These fibrils are short and even, and cannot cause pilling. Secondary fibrillation produces change in hand as well as appearance.

Other than the mechanical effect, the factors that increase fibrillation are (Kasahara Katsuji et.al 2003) *low yarn twist, open structure, high temperature, alkaline* p^H, *low liquor ratio,* etc. On the other hand, the factors that decrease fibrillation include reduced mechanical action, use of crease mark reducing agents, singeing before or after dyeing, cellulose enzymatic treatment and finishing with resins.

2.6.1 Constraints

On another hand if the fibrillation is not properly controlled means following problem will happen (J. Richard Aspland);

- Crease marks in dyed goods, graying may appear.
- Recurrent fibrillation could spoil the goods during consumer's washing.
- Pills would be entangled from badly fibrillated fibers.

2.6.2 Reason for Fibrillation

Lyocell fiber shows some key advantageous characteristics over other cellulosic fibers, for instance, a high dry and wet tenacity and during high wet state. It has been concluded from previous works that this is due to the spinning process, which causes the formation longer, and more oriented crystalline regions and smaller but more oriented amorphous regions in the fiber structure (R. S. Blackburn 2005).

This fibrillar structure is responsible for the high fiber tenacity but low lateral cohesion, especially when subjected to mechanical stress in the swelled state. Even though the fibrillation characteristic has been shown to be advantageous in applications when a peach-skin appearance of the surface is required, but it has been proven to be disadvantageous for some other applications, such as the launder ability of the product and difficulty to control the uniformity of color uptake during dyeing.

Primary fibrillation of Fibers ends at surface of the fabric:

- Steaming of fibers in spinning
- Spin finish
- Removal of fibrillated surface fibers
- Dyeing, Pad Batch or Exhaust on jet: Additional removal of fibrillated surface fibers.

Generation of **secondary fibrillation** at fabric surface:

- Treatment on tumbling machine with softener:
- A Fine secondary fibrillation formed by fiber splitting in exposed areas takes place.
- Finishing on Stenter.

2.6.3 Controlling of fibrillation

These run from controlling spinning parameters (spinneret size and temperature, draw ratio, etc.) to air gap conditions and after treatment of the fiber. Commercially, it was found practical to treating with alkalis and dyeing with some special poly-functional reactive dyes to control the degree of fibril ability (R. S. Blackburn 2005).

2.6.4 Problem while wetting the fiber

N –Methylmorpholine – N –oxide is commercially available in the form of several hydrates. The active solvent moiety of NMNO is its N – O appendage with its strong dipolar character. The oxygen of this group is able to form one or two hydrogen bonds with hydroxylated substances such as water and alcohols (Yi-Jun Pan, Chien-Kuo Yen 2008).

Similar hydrogen bonding is also believed to occur with cellulose. When the dissolution of cellulose occurs in the presence of water, there is a competition between water and cellulose for the NMNO molecules which seem to prefer water to cellulose (Wangsun Zhang, Satoko Okubayashi And Thomas Bechtold 2003). This explains why cellulose solutions can be prepared only with compositions poor in water. Thus with 13.3% of water, NMNO forms a monohydrate that readily dissolves cellulose; with 23.5% water it forms a dehydrate that will not dissolve cellulose.

Apparently, some water is needed to open the internal pores of cellulose; but too much water gets between the N-O bond of amine oxide and cellulose, thereby preventing solution.

2.6.5 Fibrillation Measurement

Many possible solutions have been proposed to modify the fibrillation structure and hence the fibril ability of the Lyocell fiber. These run from controlling spinning parameters (spinneret size, temperature, and draw ratio, etc.) to air gap conditions and after treatment of the fiber. Commercially, it was found practical to treat the finished fiber with cross-linking agents or some special poly-functional reactive dyes to control the degree of fibril ability. Fibrillation measurements are normally presented in terms of fibrillation index. FI, which is the sum of the fibril lengths ($\sum l$), divided by the fiber length (L) (W. Udomkichdecha et.al 2002)

$$FI = \sum l / L$$

The FI value is only simple measurement assessed through optical micrographs under the restriction of q two dimensional photographs. In fact, fibrillation of a Lyocell fiber occurs all around the fiber skin and thus a more accurate measurement should be carried out to take account of the third dimension (Bates I, Mauchru E, Phillips DAS, Renfrew AHM, Su Y, Xu J 2004).

However, there are still some complications in proposing any alternative model for calculating the fibrillation index in a three-dimensional photograph. Therefore, predicting the fibrillation ability by some other physical parameters such as these parameters include birefringence, intrinsic viscosity and relative crystallinity.

2.6.6 Defibrillation

Defibrillation usually precedes dyeing when a peach-skin effect is desired. However, for a totally clean surface, defibrillation is done after the dyeing stage. The primary fibrillation often referred as pre fibrillation or Meta fibrillation is understandably inevitable in normal fibrilling type of Lyocell fibers (Chae D. W., Choi K. R. and Kim B. C. 2003) .

Removal of the fibrils so generated during this process is absolutely imperative and this is done by using a treating with various alkali and followed by dyeing with poly-functional reactive dyes. Defibrillation is often done in the same machine in which pre-fibrillation occurs. The fabric treated with alkali and a poly functional reactive dye help to degrade the protruding fibrils along the surface of the fiber (A.Georgieva, D.Pishev 2001), softens the fabric, removes fluff and finally gives an even effect.

Basically fibrillation can be controlled by changing the various spinning parameters *(Nicolai M., Nechwatal A. and Mieck K.- P. 1998)* such as *spinneret size, temperature, draw ratio, air gap conditions and after treatment* of the fiber. Commercially, it was found any one of the following methods used for control the degree of fibrillability *(R. S. Blackburn 2005)*. Removal of the fibrils is absolutely imperative and this is done by using any one of the following methods *(Nechwatal A., Nicolai M. and Mieck K.- P. 1996),*

1. *Treating with various alkali's*
2. *Dyeing with poly functional reactive dyes*
3. *Enzymatic treatments*

2.7 TREATING WITH VARIOUS ALKALI'S

The pre-treatment is most important stage in chemical processing of different cellulose fibers and blends is response of these fibers to treatments involving alkali at different concentrations. During scouring, mercerization and dyeing with reactive dyes, sodium hydroxide or sodium carbonate is normally used. It is well known that the fibrillation tendency of Lyocell fibers is related to swelling state. In view of this, it is necessary to examine the effect of different types of alkali (Sodium hydroxide (Na OH) (Goswami P, Blackburn RS, El-Dessouky HM, Taylor J, White P 2009), Lithium hydroxide *(Li OH),* Potassium hydroxide (K OH), Tetra methyl- ammonium hydroxide *(TMAH)* at room temperature on Lyocell fibers. The effect of sodium hydroxide treatment (Joonseok Koh 2005) on crystallinity in the cellulose II of lyocell, modal and viscose, and followed this with a separate study comparing lyocell, acid-hydrolyzed lyocell, and standard crystalline cellulose II Recently[19], *Goswami et* *al. 2009 have observed* that sodium hydroxide treatment causes the density, orientation and crystallinity of lyocell fiber to decrease with increasing sodium hydroxide concentration, and that the greatest change in fiber properties occurs between 3.0 and 5.0 mol dm^{-3} NaOH. This was attributed to the onset of formation of sodium (Na)-cellulose II at 3.0 mol dm^{-3} NaOH; a fully formed Na-cellulose II structure was observed above 6.8 mol dm^{-3} NaOH. The work concluded that formation of Na-cellulose II causes plasticization of the lyocell fibers as both inter- and intra-molecular hydrogen bonds are broken by these higher sodium hydroxide concentrations. During the plasticization state of lyocell can be caused to reduce the fibrillation tendency up to 40 %. Lyocell is subjected to treat with Tetra methyl-ammonium hydroxide (TMAH) will reduce 60% of fibrillation tendency compare to other alkalis.

The critical degree of swelling for lyocell fiber with no fibrillation was 0.45 cm3/g in ethanol/water mixture. The fibrillation was retarded with alkali treatment in aqueous NaOH and KOH/ Li OH, solutions at concentrations between 3.0 and 7.0 mol/l, and minimized at 5.0 mol/l where the uniform reorganization of macrofibrils was observed with scanning electron microscope. The fibril number of lyocell fiber treated in trimethylammonium hydroxide was enhanced with increasing concentration and weight loss. The fibrillation was retarded by crosslinking with 1,3-dimethylol-4,5-dihydroxyethylene urea and by treatment with amino functional polysiloxane accompanying decrease in water retention capacity.

2.8 POLY FUNCTIONAL REACTIVE DYE

Generally additional reactive groups do offer the important benefit of potentially increasing the fixation of a dye. This parameter is obviously central in determining the practical color value of a particular product (J. M. Taylor, M. J. Bradbury and S. Moorhouse 2001). Covalent bond formation and hydrolysis take place concurrently during the dyeing of cellulose with a reactive dye. Clearly if the dye has only one reactive group, hydrolyzed product can no longer take part in the dyeing. However, if the dye is Poly-functional, i.e. contains more than four reactive groups, a further opportunity exists for fixation (A Hunter M Renfrew 1999).

Specific multifunctional reactive dyes are reported to have favorable effect on fibrillation behavior of Lyocell fiber (Kasahara Katsuji et al 2003). The cross linking of reactive groups of these dyes with adjacent cellulose chains provides an opportunity to reduce fibrillation during wet processing. Certain reactive dyes, which have at least two reactive centers, can form a covalent bond with two adjacent cellulose molecules. It is also believed that the presence of several reactive groups is not alone sufficient to produce this effect, but very specific molecular constitution and properties are also required to get the said effect.

As Lyocell is a cellulosic fiber, it can be dyed with colors normally used on cotton. Compared to unmercerized cotton & lyocell except with a few *reactive, vat, and direct dyes* to produce heavier depth by exhaust methods. But mostly reactive dyes are used for achieving dual functions such as dyeing and reducing degree of fibrillation (Kasahara Katsuji et al- 2003)

Reactive dyes were introduced 40 years ago, today modern dyestuffs with several reactive groups, which were originally developed for higher wet fastness and better bath exhaustion. Fundamentally bilateral reaction provides the opportunity of cross linking cellulose molecules. The excepted crosslinking in cellulose molecules will be developed. This effect is of particular interest of fibers with high fibrillation. In the case where ploy functional reactive dyes can cause more crosslinking reaction with cellulose and followed by reduce the degree of fibrillation, it stimulate to increase the wet abrasion resistance when compare to bi-functional or tri functional reactive dyes.

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Specific multifunctional reactive dyes are reported to have favorable effect on fibrillation behavior of Lyocell fiber (Rohrer C. Retzl P. and Firgo H. 2001). The cross linking of reactive groups of these dyes with adjacent cellulose chains provides an opportunity to reduce fibrillation during wet processing. Certain reactive dyes, which have at least two reactive groups, can form a covalent bond with two adjacent cellulose molecules. It is also believed that the presence of several reactive groups is not alone sufficient to produce this effect, but very specific molecular constitution and properties are also required to get the said effect.

2.9 ENZYMATIC TREATMENTS

Fibrillation can be removed by using of specific cellulase enzymes. These need to be carefully controlled, but are very effective at polishing the fabric surface to remove any un acceptable fibrillation *(Nechwatal A., Nicolai M. and Mieck, K.- P. 1996)*. Enzymes will not prevent the recurrence of fibrillation of fibers but, in conjunction with optimum processing procedures. But the result is not good when comparing with previous methods, so this method not suitable for de fibrillation of lyocell fiber.

2.10 EFFECT OF FIBRILLATION ON PILLING TENDENCY OF LYOCELL FIBER

Pilling is a phenomenon exhibited by fabrics formed from spun yarns Fink. (H.-P., Weigel P., Purz H. J. and Ganster J. 2001)' Pills are masses of tangled fibers that appear on fabric surfaces during wear or laundering. Fabrics with pills have an unsightly appearance and unpleasant handle. Loose fibers are pulled from yarns and are formed into spherical balls by the frictional forces of abrasion. These balls of tangled fibers are held to the fabric surface by longer fibers called anchor fibers. When fabrics are made from polyester, nylon, lyocell spun yarns, however, the stronger anchor fibers are not easily broken and the pills that are formed are not released quickly from the fabric (Goswami P, Blackburn RS, El-Dessouky HM, Taylor J, White P (2009).

In the swollen state lyocell has an extensive fibrillation tendency owing to linear high crystalline fibrillar morphology (Nechwatal A., Nicolai M. and Mieck, K.-P. 1996.). The fibrillation tendency of lyocell enables this fiber to be used in specific finishing effects such as *peach skin, silk touch and soft denim.* On the other hand, the fibrillations induce e.g. *rope marking defect in hank finishing, graying of dyed fabrics and a change of handle* of clothes that spoils garments features. Many reports on the morphological structure of man-made cellulosic fibers and their treatment with crosslinking agents have been published (Fink. H.-P., Weigel P., Purz H. J. and Ganster J. 2001). Fibrillation mostly leads to pilling and therefore spoils fabric appearance and touch.

Pill formation is a common problem mainly in knitted fabrics made not only from synthetic fibers but also from natural fibers, man-made cellulosic's and their blends because no consumers accept the undesirably pilled garments. There have been many studies about pilling mechanism for knitted fabrics, which described influences of selected fiber properties e.g. tensile strength, elongation, bending rigidity, fiber count, shape of fiber cross-section and friction on the pilling phenomenon. Those models were, however, established for dry conditions but not for processes including wet condition e.g. laundry. Man-made cellulosic fibers are hygroscopic materials and their structures of fiber, yarn and fabric dramatically change by swelling with polar solvent such as water (Okubayashi S., Griesser U. and Bechtold T. 2004 and Brauneis F. and Eibl M. 1998). In the present study, a pilling mechanism including fibrillation and fuzz formation in dry and wet states is discussed and concepts to achieve high durable lyocell textiles against fibrillation and pilling are suggested (Rohrer C. Retzl P. and Firgo H. 2001).

Firstly a fiber end comes out from the inside of a yarn by a mechanical abrasion during drying treatment, which induces the fuzz (1). The fuzz fiber is swollen with washing treatment and gets softer as show in (2). The swollen soft fiber is easily fibrillated by mechanical abrasion during Washing and Drying treatments (3) and then tangled each other, which develops pilling (4). The fibrillation hardly occurs in dry state (9). Some swollen fuzz would lead to pilling without fibrillation as indicated in (5). The inducement of pill formation from fuzz is significantly hindered without wetting (6). Less degree of fuzz is formed when the fiber is swollen in wet state as shown in (7). After a certain times of Washing & Drying treatments, the fiber/fiber friction in dry gets higher which results suppress of fuzz formation in dry state. Increase in fiber/fiber friction in dry state, decrease in degree of swelling might lower tendency of pill formation as well as fibrillation. *As shown in Figure 2.9,* the fibrillation plays an important role in pill formation that is significantly affected by fiber swelling (M. Nicolai et al 1996).

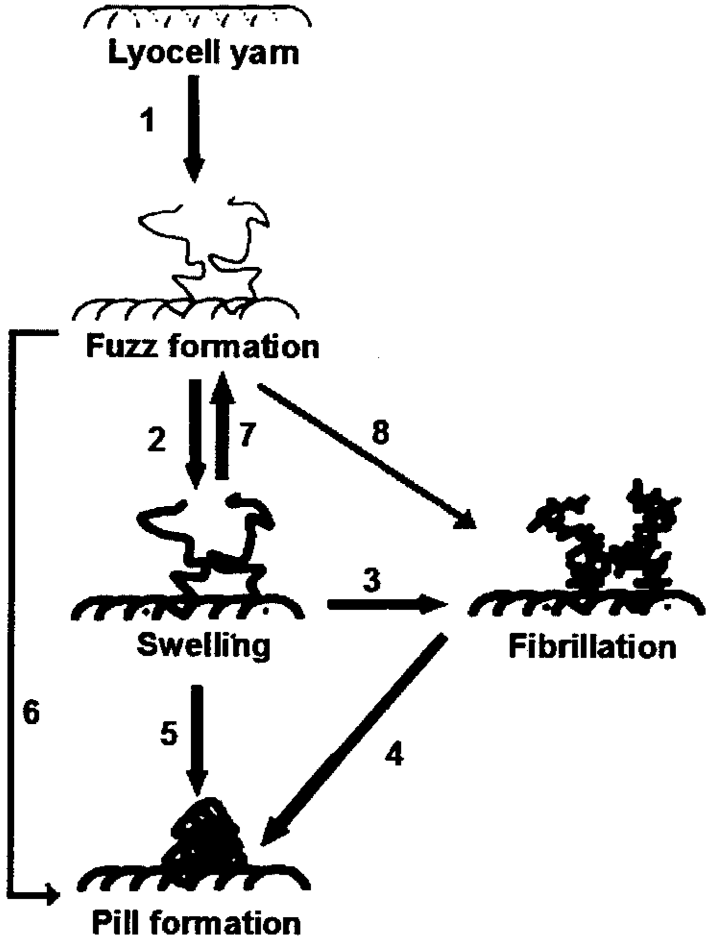


Figure 2. 9 A schematic mechanism of pill formation during Washing & Drying treatments. Arrows indicate the fuzz formation (1, 7), the swelling (2), the fibrillation (3, 8) and the pill formation (4, 5, 6)

CHAPTER 3

MATERIALS AND METHODS

3.1 MATERIALS

3.1.1 Fabric Particulars

The pure 100 % lyocell fabric were taken for this present work, first the fabric were scoured to remove the unwanted substances by using different alkalis and different concentration and at boil for one hour. The same fabric was bleached using hydrogen peroxide at 1:8 m:l ratio and at boil for one hour, then through washing & peroxide killer treatment were given to get a ready for dyeing fabric.

Table 3.1 Particulars of Lyocell Fabric

S.no	Descriptions	Properties
1	Course count	24 Ne
2	Wales count	24 Ne
3	Loop length	2.4 mm
4	Course per Inch	36
5	Wales per Inch	56
6	Weight (GSM @ Grey Stage)	150
7	CSP	3360

3.1.2 Chemicals

The following chemicals will be used for this study;

i. Poly functional Reactive Dyes,
ii. Sodium hydroxide (Na OH),
iii. Lithium hydroxide (Li OH),
iv. Potassium hydroxide (K OH),
v. Tetra methyl- ammonium hydroxide (TMAH)
vi. Hydrogen peroxide
vii. Deminarilizing agent

Above Chemicals should be the grade of "LR"

3.2. METHODOLOGY

3.2.1 Sodium Hydroxide Treatment for Lyocell

Fabric formation (Knitting)

↓

Alkali treatments *(NaOH-1, 2, 3 Gpl)*

↓

Bleaching *(H_2O_2- 1.75 Gpl)*

($95\,^0$ C, for one hour)

↓

Peroxide Killing Process

(Peroxide Killing agents- 0.5 %

Hot wash for 15 Mins)

↓

Neutralizing

(CH_3COOH- 0.8 Gpl, warm Wash for 20 min)

↓

Determine the physical properties

(Abrasion Resistance, Pilling Tendency, Abrasion Resistance)

& SEM Analysis

Figure 3.1 Flow Chart on Sodium Hydroxide Treatment for Lyocell

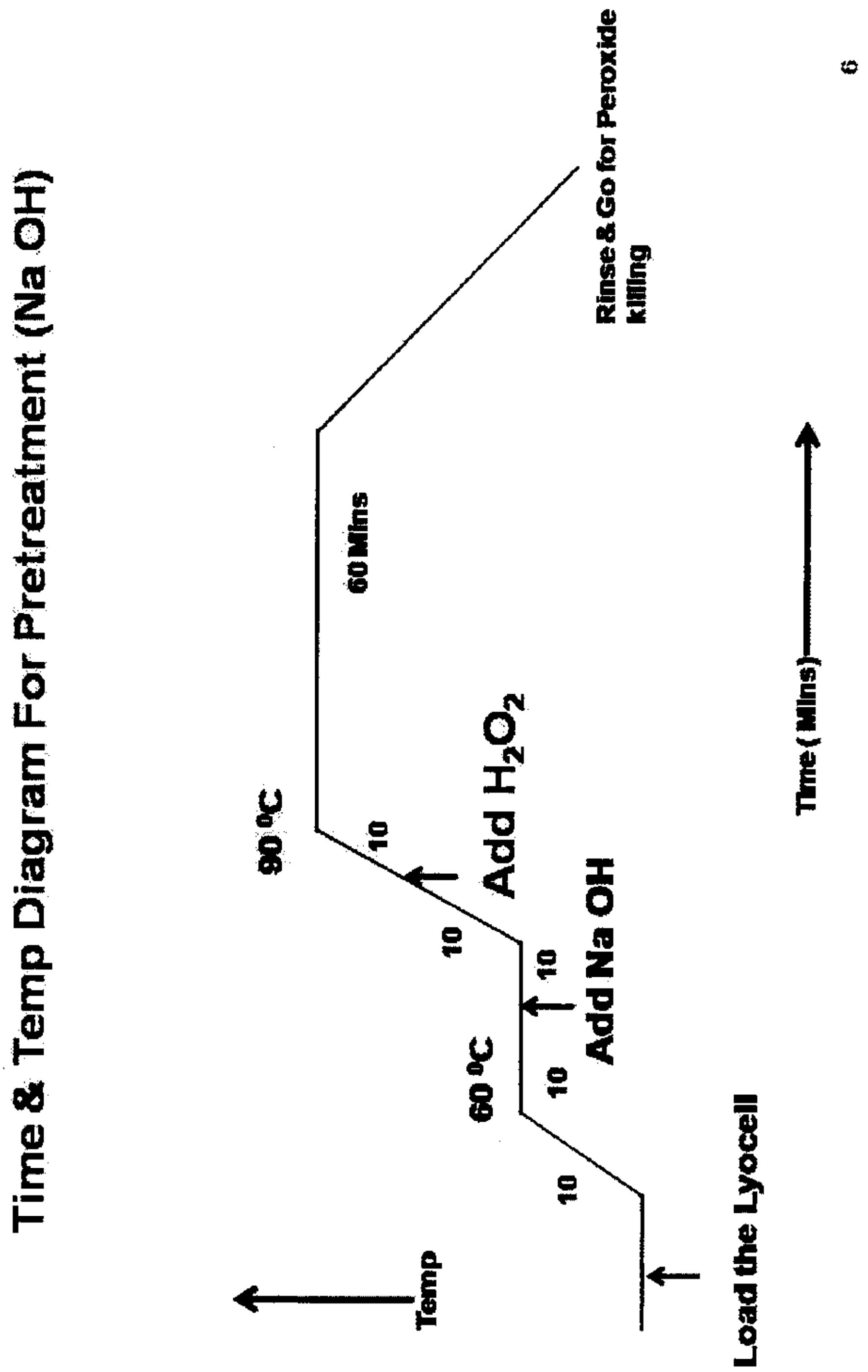


Figure 3.2 Time and Temperature diagram for Sodium Hydroxide treatment on lyocell

3.2.2 Potassium Hydroxide Treatment for Lyocell

Fabric formation (Knitting)

↓

Alkali treatment *(K OH-1, 1.5, 2 Gpl)*

↓

Bleaching ***(H_2O_2- 1.75 Gpl)***

(95 ^{0} C, for one hour)

↓

Peroxide Killing Process

(Peroxide Killing agents- 0.5 %

Hot wash for 15 Mins)

↓

Neutralizing

(CH_3COOH- 0.8 Gpl, warm Wash for 20 min)

↓

Determine the physical properties

(Abrasion Resistance, Pilling Tendency,)

& SEM Analysis

Figure 3.3 Flow Chart on Potassium Hydroxide Treatment for Lyocell

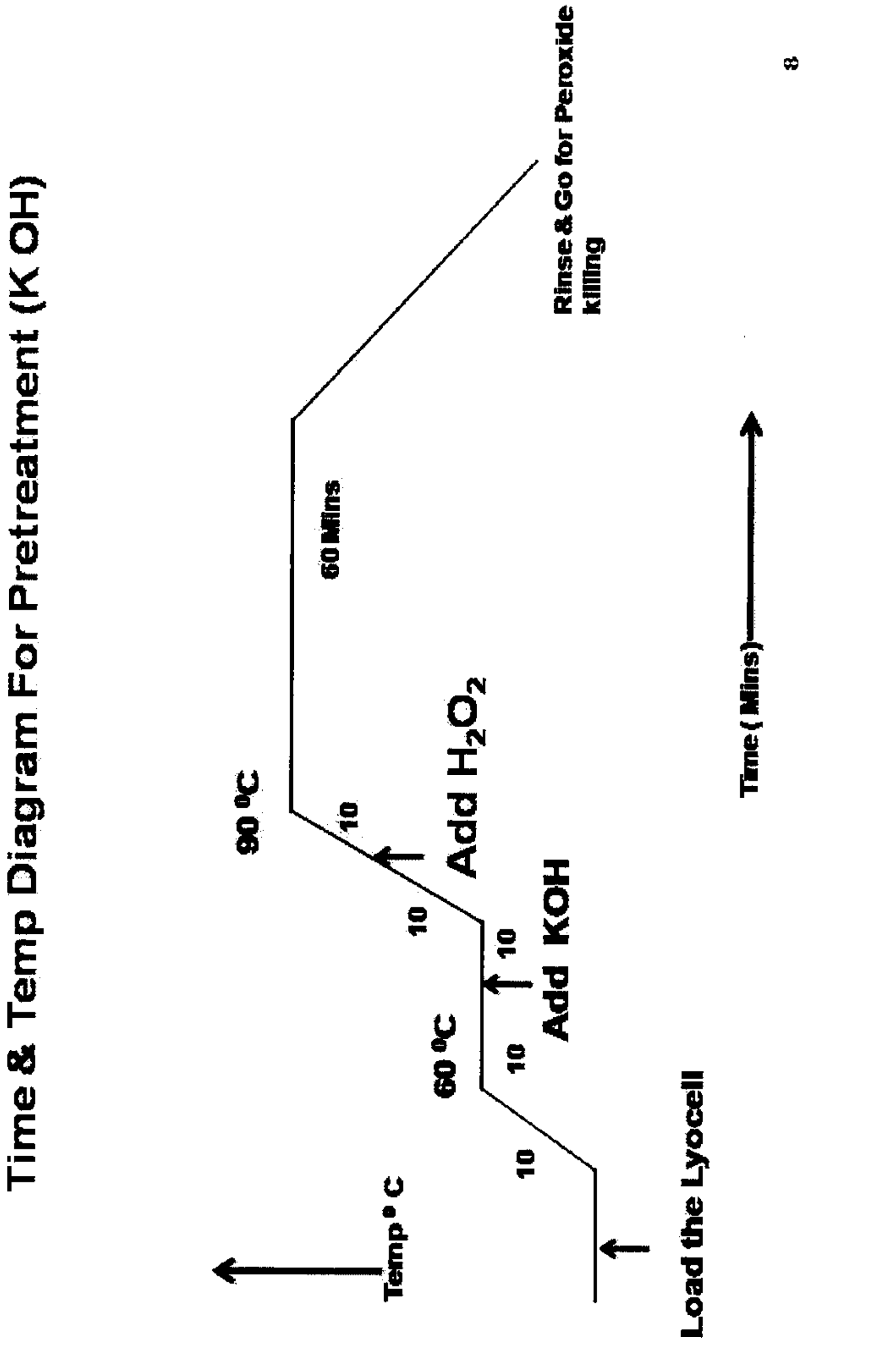


Figure 3.4 Time and Temperature diagram for Potassium Hydroxide treatment on lyocell

3.2.3 Lithium Hydroxide Treatment for Lyocell

Fabric formation (Knitting)

↓

Alkali treatment *(Li OH- 0.5, 1, 1.5 Gpl)*

↓

Bleaching *(H_2O_2- 1.75 Gpl)*

(95^{0} C, for one hour)

↓

Peroxide Killing Process

(Peroxide Killing agents- 0.5 %

Hot wash for 15 Mins)

↓

Neutralizing

(CH_3COOH- 0.8 Gpl, warm Wash for 20 min)

↓

Determine the physical properties

(Abrasion Resistance, Pilling Tendency,)

& SEM Analysis

Figure 3.5 Flow Chart for Lithium Hydroxide Treatment of Lyocell

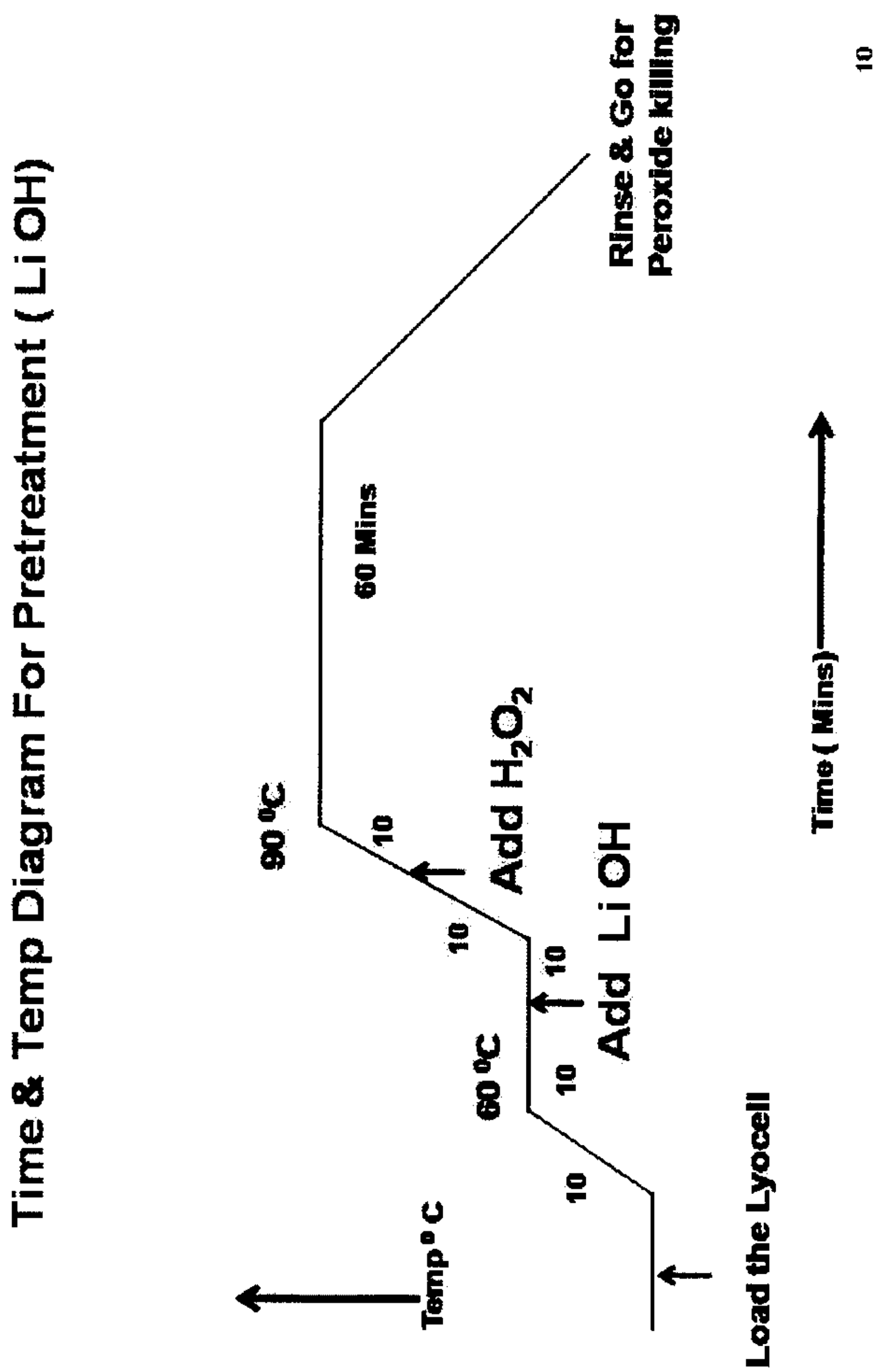


Figure 3.6 Time and Temperature diagram for Lithium Hydroxide treatment on lyocell

3.2.3 Tetra methyl- ammonium hydroxide Treatment for Lyocell

Fabric formation (Knitting)

⇓

Alkali treatment ***(Tm AH - 0.25, 0.5, 1 Gpl)***

⇓

Bleaching ***(H_2O_2- 1.75 Gpl)***

(95 ^{0} C, for one hour)

⇓

Peroxide Killing Process

(Peroxide Killing agents- 0.5 %

Hot wash for 15 Mins)

⇓

Neutralizing

(CH_3COOH- 0.8 Gpl, warm Wash for 20 min)

⇓

Determine the physical properties

(Abrasion Resistance, Pilling Tendency)

& SEM Analysis

Figure 3.7 Flow Chart on Tetra methyl- ammonium hydroxide Treatment for Lyocell

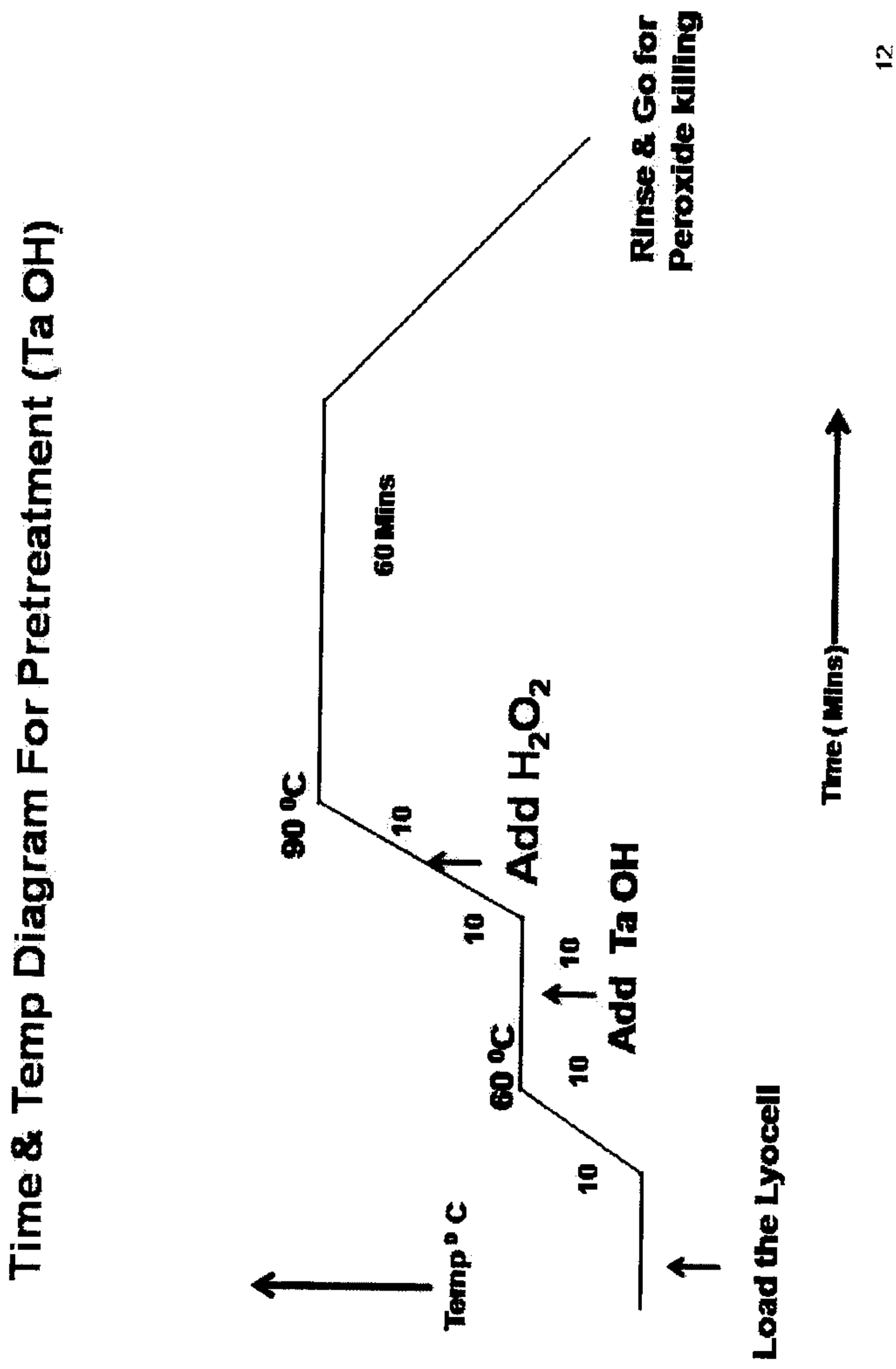


Figure 3.8 Time and Temperature diagram for Tetra methyl- ammonium hydroxide treatment on lyocell

3.2.5 Alkali treatments and Dyeing (H.E Method) of lyocell

Fabric formation (Knitting)

↓

Alkali treatments

(NaOH, K OH, LiOH & Tm AH at Three different concentration)

↓

Bleaching (H_2O_2)

H_2O_2- 1.75 Gpl, 95 ^{0} C, for one hour)

↓

Hot Wash (Residual Peroxide removing process)

(15 Mins for 80 ^{0} C)

↓

Neutralizing

(0.5 % CH_3 COOH) & 20 Mins for 60 ^{0} C

↓

Dyeing of lyocell with poly functional reactive dyes (H.E Method)

↓

Determine the physical & chemical properties

(Abrasion Resistance, Pilling Tendency, Color strength, Fastness properties)

& SEM Analysis

Figure 3.9 Flow Chart on Alkali treatments and Dyeing (H.E Method) of lyocell

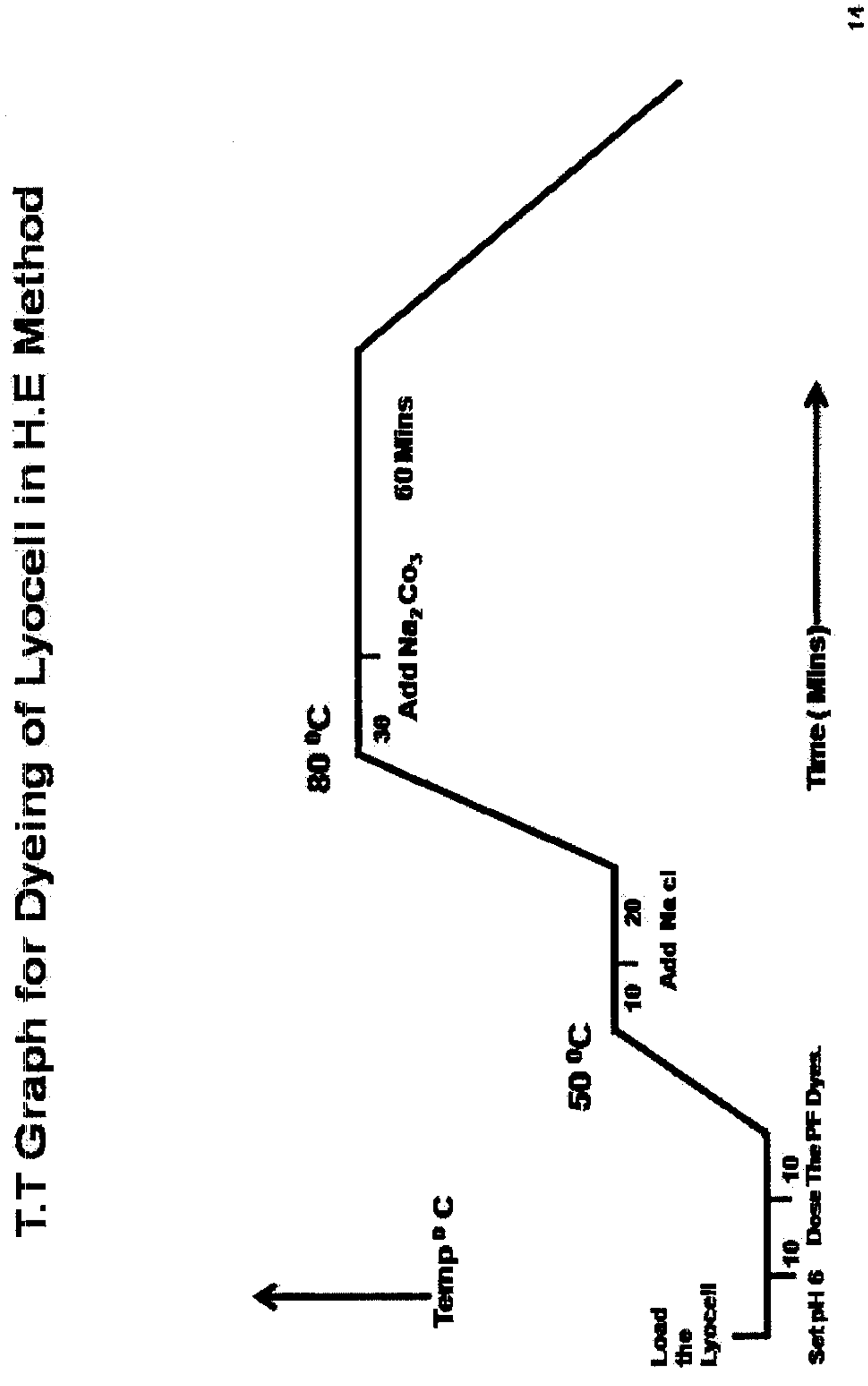


Figure 3.10 Time and Temperature diagram for Dyeing (H.E Method) of lyocell

3.2.6 Alkali treatment and Dyeing (Migration Method) of lyocell

Fabric formation (Knitting)

⇩

Alkali treatments

(NaOH, K OH, LiOH & Tm AH at Three different concentrations)

⇩

Bleaching (H_2O_2)

H_2O_2- 1.75 Gpl, 95 ^{0} C, for one hour)

⇩

Hot Wash (Residual Peroxide removing process)

(15 Mins for 80 ^{0} C)

⇩

Neutralizing

(0.5 % CH_3 COOH) & 20 Mins for 60 ^{0} C

⇩

Dyeing of lyocell with poly functional reactive dyes (Migration Method)

⇩

Determine the physical & chemical properties

(Abrasion Resistance, Pilling Tendency, Color strength, Fastness properties)

& SEM Analysis

Figure 3.11 Flow Chart on Alkali treatment and Dyeing (Migration Method) of lyocell

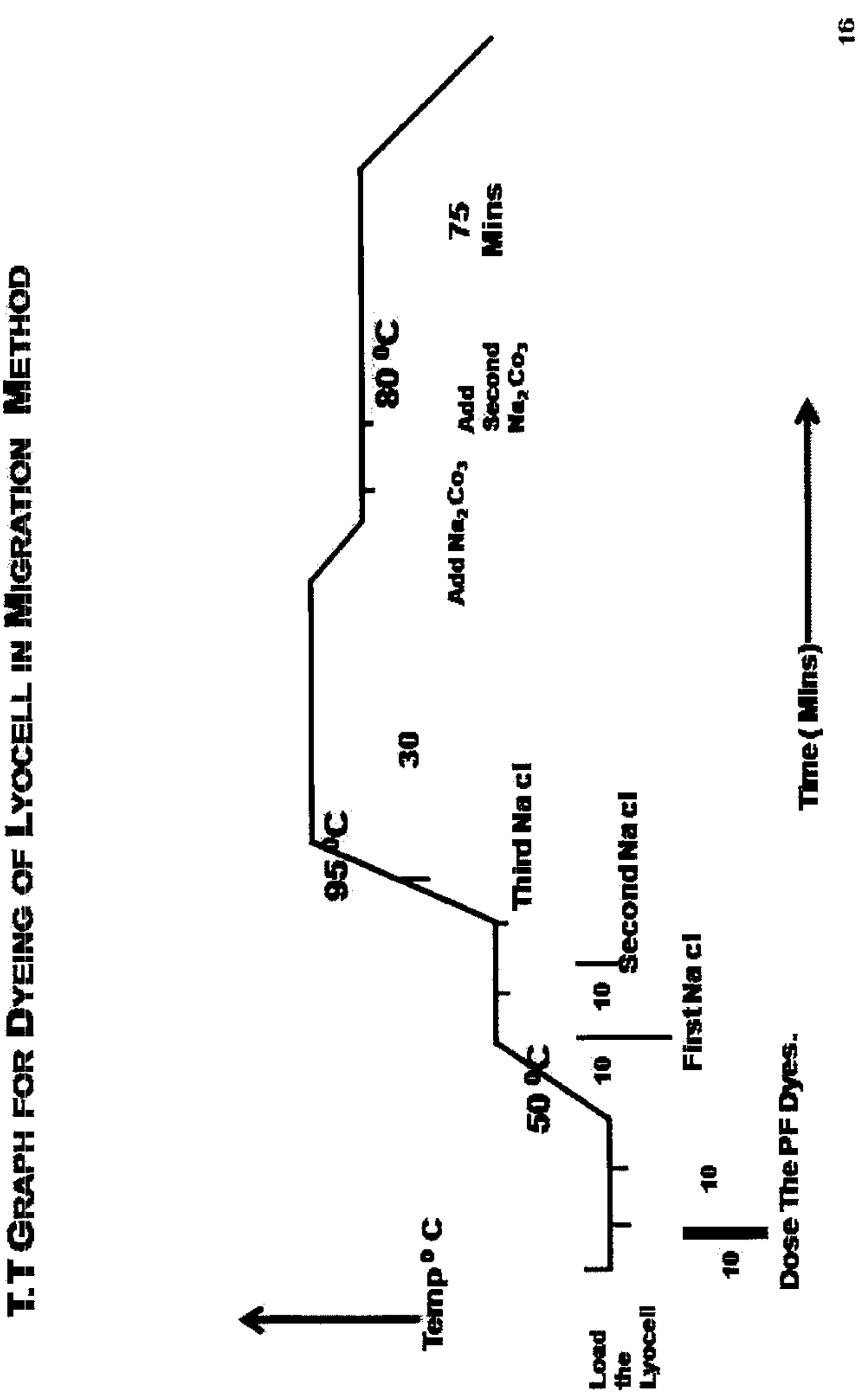


Figure 3.12 Time and Temperature diagram for Dyeing (Migration Method) of lyocell

3.3 TESTING METHODS

The following tests were carried out for treated (Alkali and Poly functional Reactive dyes) lyocell,

1. Pilling Resistance (ISO 12945-1- 1999)
2. Abrasion Resistance (ISO 12947-2- 1999)
3. SEM Analysis for Fibrillation
4. Color Strength (AATCC 182:2005)
5. Color Fastness to Water (AATCC 107-2002)
6. Color Fastness to Crocking (AATCC 008-2005)
7. Color Fastness to Light (AATCC 016-2004)

3.3.1 Assessment of Pilling Resistance

The amount of pilling that appears on a specific fabric in actual wear will vary with the individual wearer and the general conditions of use. Consequently garments made from the same fabric will show a wide range of pilling after wear which is much greater than that shown by replicate fabric specimens subjected to controlled laboratory tests. Finishes and fabric surface changes may exert a large effect on pilling. Therefore, with some fabrics, it may be desirable to test before as well as after laundering or dry cleaning or both. After rubbing of a fabric it is possible to assess the amount of pilling quantitatively either by counting the number of pills or by removing and weighing them. However, pills observed in worn garments vary in size and appearance as well as in number. The appearance depends on the presence of lint in the pills or the degree of color contrast with the ground fabric. These factors are not evaluated if the pilling is rated solely on the number or size of pills. Furthermore the development of pills is often accompanied by other surface changes such as the development of fuzz which affect the overall acceptability of a fabric. It is therefore desirable that fabrics tested in the laboratory are assessed subjectively with regard to their acceptability and not rated solely on the number of pills developed.

Counting the pills and/or weighing them as a measure of pilling is very time consuming and there is also the difficulty of deciding which surface disturbances constitute pills. The more usual way of evaluation is to assess the pilling subjectively by comparing it with either standard samples or with photographs of them or by the use of a written scale of severity

3.3.2 Assessment of Abrasion Resistance

The evidence concerning the various factors that influence the abrasion resistance of fabrics is contradictory. This is because the experiments have been carried out under widely different conditions in particular using different modes of abrasion. Two approaches have been used to assess the effects of abrasion:

1. Abrade the sample until a predetermined end-point such as a hole, and record the time or number of cycles to this.
2. Abrade for a set time or number of cycles and assess some aspect of the abraded fabric such as change in appearance, loss of mass, loss of strength change in thickness or other relevant property.

The first approach corresponds to most people's idea of the end point of abrasion but the length of the test is indeterminate and requires the sample to be regularly examined for failure in the absence of a suitable automatic mechanism. This need for examination is time consuming as the test may last for a long time. The second approach promises a more precise measurement but even when the sample has rubbed into a hole the change in properties such as mass loss can be slight. However none of the above assessment methods produces results that show a linear or direct comparison with one another. Neither is there a linear relationship between successive measurements using any of these methods and progressive amounts of abrasion.

Abrasion resistance for hosiery

This test makes use of a modified specimen holder for the Martindale abrasion tester, which stretches the knitted material thus effectively accelerating the test. The holder takes a standard size 38mm diameter sample which is held to size by a pinned ring. A flattened rubber ball is pushed through the sample as the holder is tightened thus stretching it. The holder is then mounted on the Martindale with a 12kPa weight and the test carried out as normal. The sample is inspected at suitable intervals until a hole appears or the material develops an unacceptable level of thinning.

Martindale

This apparatus is designed to give a controlled amount of abrasion between fabric surfaces at comparatively low pressures in continuously changing directions. The results of this test should not be used indiscriminately, particularly not for comparing fabrics of widely different fiber composition or construction. In the test circular specimens are abraded under known pressure on an apparatus, which gives a motion that is the resultant of two simple harmonic motions at right angles to one another. The fabric under test is abraded against a standard fabric. Resistance to abrasion is estimated by visual appearance or by loss in mass of the specimen.

Method

Four specimens each 38mm in diameter are cut using the appropriate cutter. They are then mounted in the specimen holders with a circle of standard foam behind the fabric being tested. The components of the standard holder are important that the mounting of the sample is carried out with the specimens placed flat against the mounting block. The test specimen holders are mounted on the machine with the fabric under test next to the abradant. A spindle is inserted through the top plate and the correct weight (usually of a size to give a pressure of 12kPa but a lower pressure of 9kPa may be used if specified) is placed on top of this. The standard abradant should be replaced at the start of each test and after 50,000 cycles if the test is continued beyond this number. While the abradant is being replaced it is Assessment The specimen is examined at suitable intervals without removing it from its holder to see whether two threads are broken. If the likely failure point is known the first inspection can be made at 60% of that value. The abrading is continued until two threads are broken. All four specimens should be judged individually. Average rate of loss in mass this is an alternative method of assessing abrasion resistance which requires eight specimens for the test. Two of these are abraded to the endpoint as described above and then the other pairs are abraded to the intermediate stages of ***25%, 50% and 75%*** of the end point. The samples are weighed to the nearest 1 mg before and after abrasion so that a graph can be plotted of weight loss against the number of rubs. From the slope of this graph, if it is a straight line, the average loss in mass measured in ***mg/1000 rubs*** can be determined.

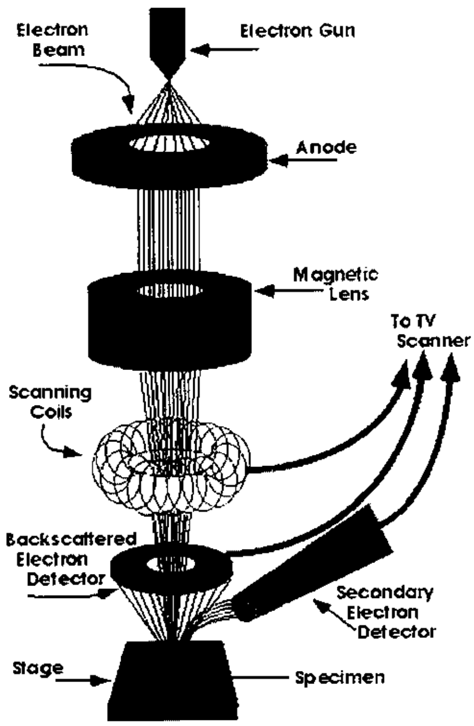
3.3.3 Scanning Electron Microscopy Analysis

In electron microscopy, electron beams are used as the radiation. The wavelength depends on the energy of the electrons, which depends on the voltage applied for their acceleration. The resolution, which depends on the wavelength of the radiation used, is as low as a few angstrom units in the most efficient electron microscope.

Electron microscopy can be performed in both reflection and transmission modes. In the reflection mode the beam is scanned over the specified area of the sample and the image is formed; thus the technique is generally known as *Scanning Electron Microscopy* (or SEM).

- In the case of *Transmission Electron Microscopy* (TEM), modern versions of the equipment also have scanning arrangements and are called *Scanning Transmission Electron Microscopes* (STEM). Furthermore, instead of just simple microscopy, these electron microscopes provide the possibility of studying various phenomena such as electron diffractions, emission of secondary electrons, the cathodoluminescence effect etc.

Figure 3.13 Scanning Electron Microscopy



An electron microscope works on the same principle as a light microscope. The beam of electrons emerging from a hot metallic cathode to a fine beam through electromagnetic lenses. The collimated beam is converged by another set of electro magnetic lenses called the 'objective' and directed onto the specimen. The beam after transmission (or reflection) converges at the focal point to form the image, which is subsequently magnified through another set of electromagnetic lenses. The final image is then formed on a fluorescent screen or can be recorded photography. In the scanning electron microscope, the fine collimated beam of electrons is deflected through a suitable scanning coil device, so that it falls on different areas of the specimen surface at different moments of time. The circuit for detection of the reflected beam is synchronized with the initial scanning coils to detect the reflected beam from each area of the specimen surface at the respective moment of time.

3.3.4 Assessment of Colour Strength

Color strength K/S was measured on Minolta Spectrophotometer. These values are calculated using the following **"KUBELKA-MUNK"** equation.

$$\textit{Colour Strength} = \frac{K/S = (1-R)^2}{2R}$$

Where **K**- Absorption co-efficient, **R**- Reflectance of the dyed sample,

S – Scattering co-efficient at the wavelength of maximum absorption.

3.3.5 Assessment of Colour Fastness

Poor color fastness in textile products is a major source of customer complaint. The fastness of a color can vary with the type of dye, the particular shade used, the depth of shade and how well the dyeing process has been carried out. Dyes can also behave differently when in contact with different agents, for instance dyes which may be fast to dry-cleaning may not be fast to washing in water. It is therefore important to test any dyed or printed product for the fastness of the colors that have been used in its decoration. There are a number of agencies that the colored item may encounter during its lifetime which can cause the color either to fade or to bleed onto an adjacent uncolored or light colored item. These factors vary with the Bend use for which the product is intended.

For instance carpets and upholstery are cleaned in a different way from bed linen and clothing and therefore come into contact with different materials. The agencies that affect colored materials include light, washing, dry-cleaning, water, perspiration and ironing. There are a large number of color fastness tests in existence which deal with these agencies and a full list will be found in the British Standard.

A further group of tests is connected to processes in manufacturing that the colored material may undergo after dyeing but before completion of the fabric, processes such as decateising or milling. Despite the fact that the list of color fastness tests is very long, most of them are conducted along similar lines so that the main differences among the tests are in the agents to whom the material is exposed. During wet treatments, such as washing and dry cleaning also adjacent undyed materials may take up color due to the transfer of dye from the original dyed material. This is known as staining. Basically for fastness property of dye depends upon the chemical structure of the colorant, dye concentration, environmental condition, nature of fiber, atmospheric contaminants, presence of foreign substances and state of dye inside the fabric. The outstanding property of the dye material is the fastness of its shade.

3.3.6 Assessment of Rubbing Fastness

Crocking relates to the property by which a textile transfers a colorant(s) from the surface of a colored yarn or fabric to another surface or adjacent area of the same fabric principally by rubbing. Crocking is evaluated using according *to AATCC Test Method 8-1996. Textile Institute (1991)* states that crocking is the rubbing fastness of dyed fabrics. Crocking or rubbing is important in both apparel and those used for upholstery. Crocking is transfer of colorant from the surface of a colored yarn or fabric to another surface or to an adjacent area of the same fabric principally by rubbing. Each of the printed material was cut to size of 20cm x 10 cm and mounted on a flat base of the crock meter. A white desized material of 5cm x 5 cm dimension was mounted on to the rubbing finger with a ring. Each sample was given ten rubs based on the standardization. The color transferred from the dyed samples to the white material was assessed using grey scale. A damp dyed sample was used for wet crocking.

3.3.7 Assessment of Light Fastness

This test measures the resistance to fading of dyed textiles when exposed to daylight. The test is of importance to the dyestuff manufacturer, the dyer and the retailer. Certain end products require a high resistance to fading because of their exposure to light during use, for example: curtains, upholstery, carpets, awnings and coatings. However, many items of apparel also require a degree of light fastness because they are exposed to light when on display, particularly in a shop window.

Light sources

The British Standard allows either daylight or xenon arc light to be used for the test.

Xenon arc B02

The xenon arc is a much more intense source of light which has a very similar spectral content to that of daylight so that the test is speeded up considerably. Because of the large amount of heat generated by the lamp an efficient heat filter has to be placed between the lamp and specimen and the temperature monitored. This is in addition to a glass filter as above to remove ultra-violet radiation.

The essence of the test is to expose the sample under test to the light source together with eight blue wool reference standards. The sample and blue standards are partly covered so that some of the material fades and some is left unfaded. A rating is given to the sample which is the number of the reference standard which shows a similar visual contrast between the exposed and unexposed portions as the specimen. This means that the specimen will be given a grade between *one* (poor light fastness) and *eight* (highly resistant to fading). If the result is in between two blue Dyeings it is given as 3-4, for example. There are two sets of blue wool reference standards in use. Those used in Europe are identified by the numerical designation 1 to 8. *They range from 1 (very low light fastness) to 8 (very high light fastness)* so that each higher numbered reference is approximately twice as fast as the preceding one.

CHAFTER-4

RESULT AND DISCUSSION

4.1 EFFECT OF PILLING TENDENCY ON ALKALI TREATED LYOCELL

Visual evaluation of the samples revealed that the pilling of fabrics after alkali treated lyocell was better than that of the one which underwent the primary fibrillation process. Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, and then observes the pilling grades. Finally the result show the pilling grades of the laundered fabric samples restrained which is treated with different alkalis. Test result shown in bellow the table no 4.1, typically pilling grade is excellent when the concentration of alkalis is increase, as well as the type of alkali used, when we used Tetra methyl- ammonium hydroxide for scouring the pilling doest formed, the grade we get it 4 (as per ISO 12945-1 standard) when the concentration of 1 gpl. As per the ISO 12945-1 standard, the grade 5 represent the outstanding pilling resistance, as well as 1 represent the poor pilling resistance,

Table 4.1 Pilling test results of Alkali treated lyocell

S. No	Alkali Treated		
	Sample code	Concentration (gpl)	Pilling Tendency
1.	--	Original sample	3
2.	A1	Na OH/1	3
3.	A2	Na OH/2	3
4.	A3	Na OH/3	3
5.	A4	K OH/1	3
6.	A5	K OH/1.5	3
7.	A6	K OH/2	3-4
8.	A7	Li OH/0.5	3
9.	A8	Li OH/1	3-4
10.	A9	Li OH/1.5	4
11.	A10	Ta OH /0.25	3-4
12.	A11	Ta OH /0.5	4
13.	A12	Ta OH /1	4

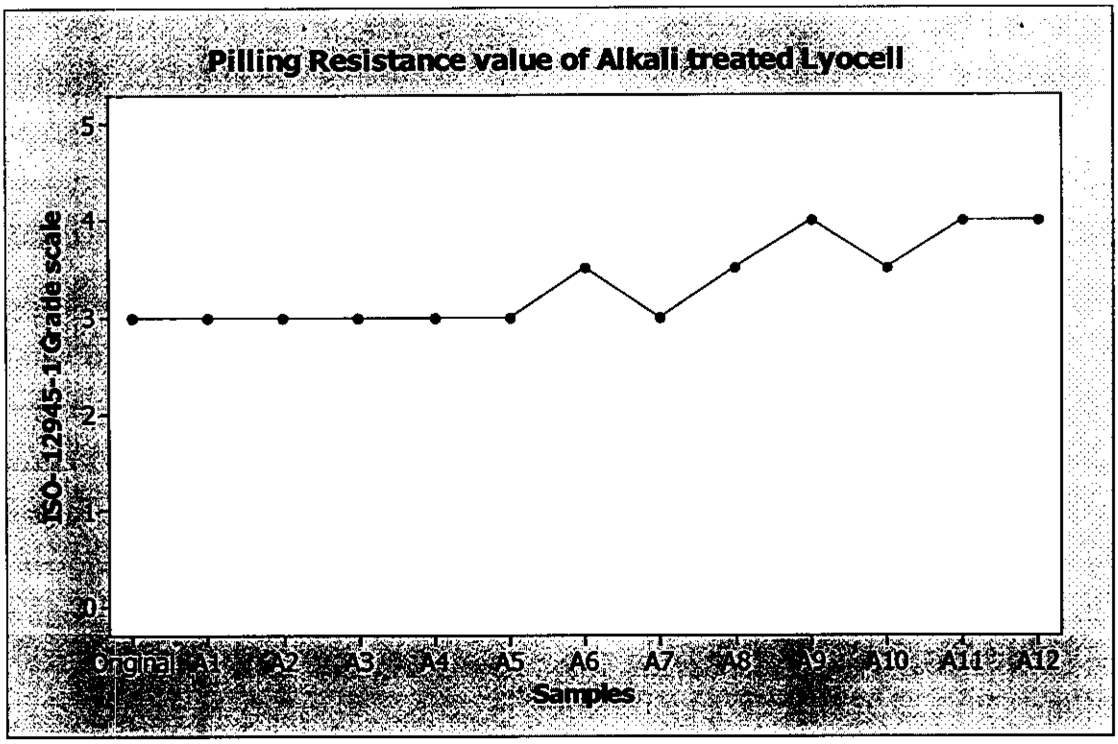


Figure 4.1 The graphical representation of the pilling test on alkali treated lyocell

4.2 EFFECT OF PILLING TENDENCY ON POLYFUNCTIONAL REACTIVE DYED (HIGH EXHAUSTION) LYOCELL

Visual evaluation of the samples revealed that the pilling of fabrics after alkali and Polyfunctional Reactive dyed lyocell was better than that of the one which conventional processed lyocell. Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, and then observes the pilling grades. Finally the result show the pilling grades of the laundered fabric samples moderate which is treated with different alkalis. The Polyfunctional reactive dyes also directly influenced to the pill formation, As per previous observed pilling grade is slightly increase when the concentration of alkalis is increase, as well as the type of alkali used, when we used Tetra methyl- ammonium hydroxide for scouring the pilling doest formed, the grade we get it 4 (as per ISO 12945-1 standard) when the concentration of 1 gpl. When we dyed the same fabric with Polyfunctional reactive dyes (H.E method) , it will increase the pilling grades, the test result shown in bellow the table no 4.2

Table 4.2 Pilling test results of on Polyfunctional Reactive dyed (H.E) lyocell

S. No	H.E Method		
	Sample code	Alkali concentration (gpl)	Pilling Tendency
1.	--	Original	2-3
2.	B1	Na OH/1/ H.E	3
3.	B2	Na OH/2 /H.E	3
4.	B3	Na OH/3 /H.E	3-4
5.	B4	K OH/1 /H.E	3-4
6.	B5	K OH/1.5 /H.E	3-4
7.	B6	K OH/2 /H.E	4
8.	B7	Li OH/0.5 /H.E	3-4
9.	B8	Li OH/1 /H.E	4
10.	B9	Li OH/1.5 /H.E	4-5
11.	B10	Ta OH /0.25 /H.E	4
12.	B11	Ta OH /0.5 /H.E	4
13.	B12	Ta OH /1 /H.E	4-5

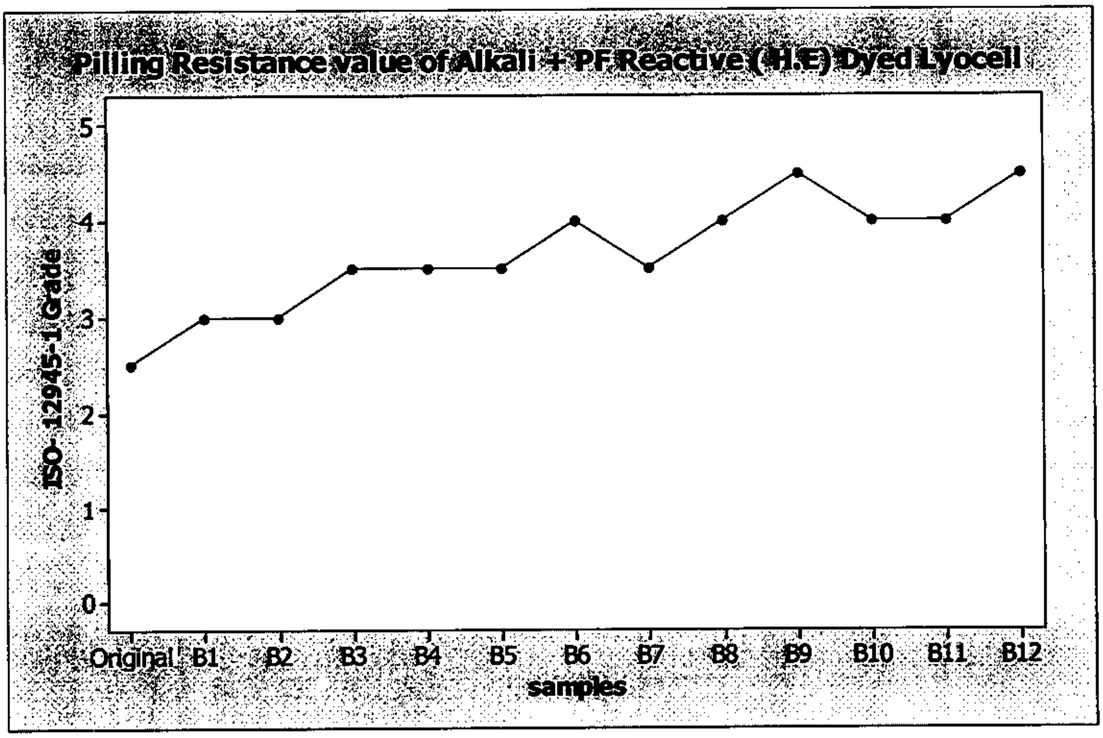


Figure 4.2 The graphical representation of the pilling test on Polyfunctional Reactive dyed (H.E) lyocell

4.3 EFFECT OF PILLING TENDENCY ON POLYFUNCTIONAL REACTIVE DYED (MIGRATION METHOD) LYOCELL

Lyocell fabric laundered ten times as per the AATCC 135-2004 standard, and then observes the pilling grades. Finally the result show the pilling grades of the laundered fabric samples moderate which is treated with different alkalis. The Polyfunctional reactive dyes also directly influenced to the pill formation, As per previous observed pilling grade is slightly increase when the concentration of alkalis is increase, as well as the type of alkali used, when we used Tetra methyl- ammonium hydroxide for scouring the pilling doest formed, the grade we get it 4 (as per ISO 12945-1 standard) when the concentration of 1 gpl, the test result shown in bellow the table no 4.3, When we dyed the same fabric with Polyfunctional reactive dyes (Migration method) it will increase the pilling grades up to 5, basically the migration method of dyeing increase the dyeability due to the higher movement of reactive dyes. Generally the migration method of Polyfunctional reactive dyes produce excellent pilling grade is due to the more no of cholorotriaznyl group formed a covalent bond with hydroxyl group of lyocell, it causes to reduce the fibrillation generation, it shows excellent pilling resistance.

Table 4.3 Pilling test results of on Polyfunctional Reactive dyed (Migration) lyocell

S.no	Migration Method		
	Sample code	Alkali concentration (gpl)	Pilling Tendency
1.	--	Original	3
2.	C1	Na OH/1/ Migration	3-4
3.	C2	Na OH/2 /Migration	3-4
4.	C3	Na OH/3 /Migration	4
5.	C4	K OH/1 /Migration	4
6.	C5	K OH/1.5 /Migration	4
7.	C6	K OH/2 /Migration	4-5
8.	C7	Li OH/0.5 /Migration	4
9.	C8	Li OH/1 /Migration	4-5
10.	C9	Li OH/1.5 /Migration	4-5
11.	C10	Ta OH /0.25 /Migration	4-5
12.	C11	Ta OH /0.5 /Migration	4-5
13.	C12	Ta OH /1 /Migration	5

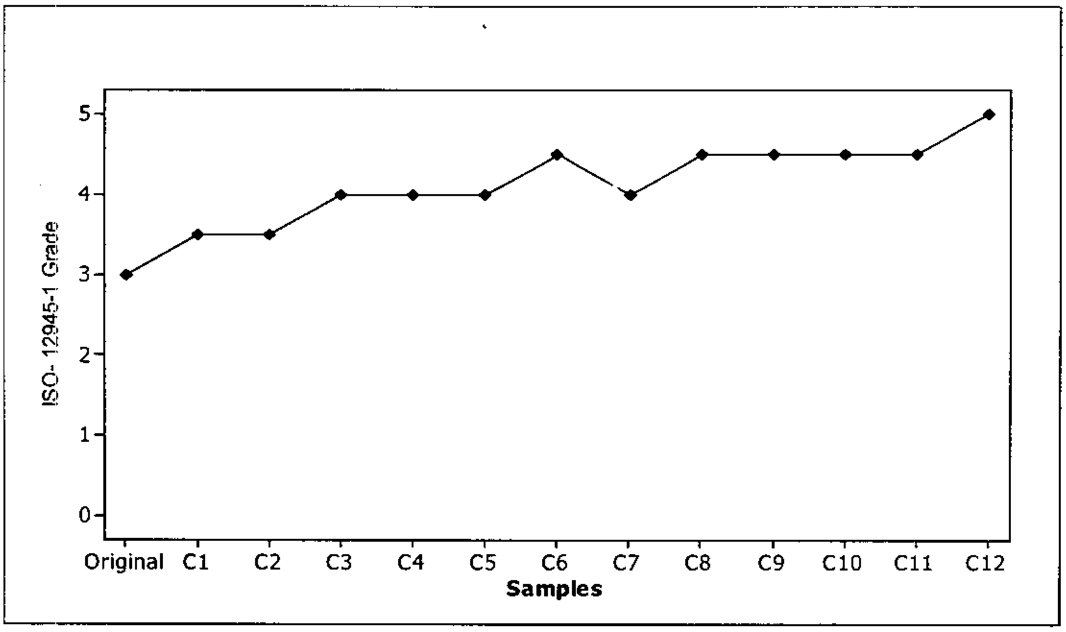


Figure 4.3 The graphical representation of the pilling test on Polyfunctional Reactive dyed (Migration) lyocell

4.4 EFFECT OF ABRASION RESISTANCE ON ALKALI TREATED LYOCELL

Abrasion is the wearing a way of any part of a material by rubbing against another surface. Adequate abrasion resistance of textile materials is essential for customer acceptance and stratification. This Martindale apparatus is designed to give a controlled amount of abrasion between fabric surfaces at comparatively low pressures in continuously changing directions.

The results of this test should not be used indiscriminately particulars not for comparing fabrics of widely different fiber composition or construction. This present study of abrasion resistance of Alkali treated lyocell fabrics was determined with a Martindale abrasion resistance tester according to ISO 12947-2- 1999 method. *For this present study,* both parameters such as concentration and type of alkalis are directly influenced the abrasion resistance of lyocell fabrics. When lyocell treated with sodium hydroxide is decrease the abrasion resistance than when we used for Tetra methyl- ammonium hydroxide for scouring. Effect of abrasion resistance on alkalis is respectively *Sodium hydroxide < Potassium hydroxide < Lithium hydroxide < Tetra methyl- ammonium hydroxide*, the test result were given in the bellow table no 4.4,

Table 4.4 Abrasion Resistance test results of on alkali treated lyocell

S.No	Alkali Treated	
	Sample code	Abrasion Resistance [illegible] Pressure 9 Kpa (End Point Found @ Rubs)
1.	Original	27200
2.	A1	24400
3.	A2	24900
4.	A3	25600
5.	A4	24700
6.	A5	25300
7.	A6	26000
8.	A7	25300
9.	A8	25900
10.	A9	26200
11.	A10	26000
12.	A11	26800
13.	A12	27400

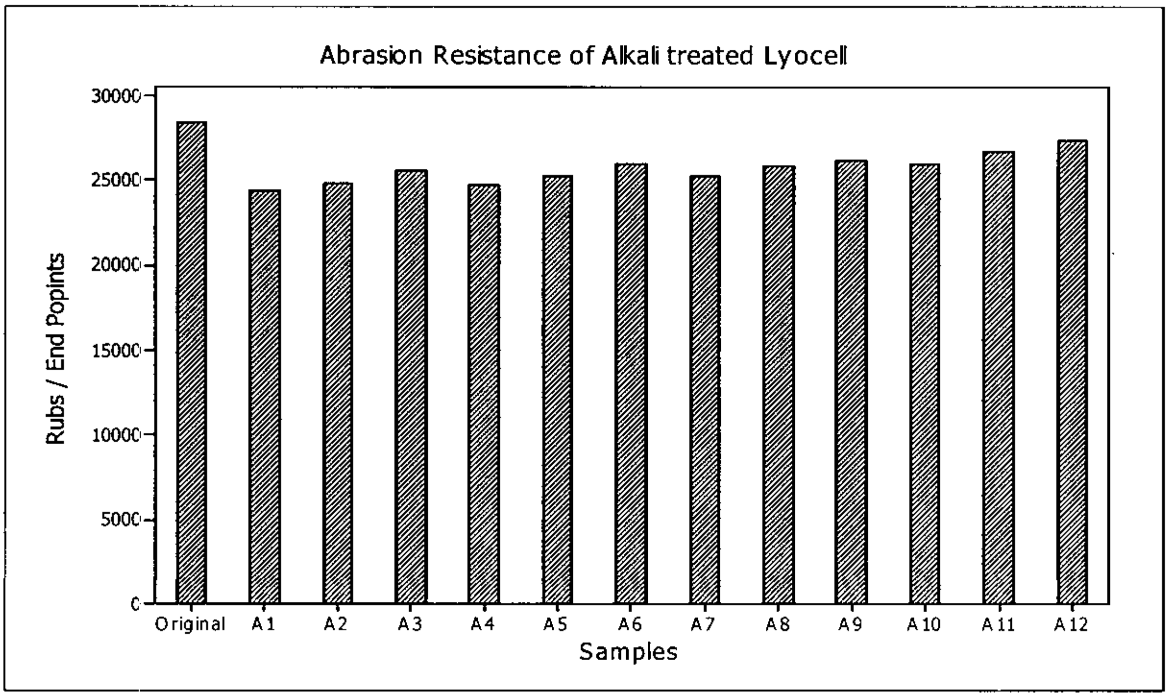


Figure 4.4 The Bar chart representation of the Abrasion resistance test on alkali treated lyocell

4.5 EFFECT OF ABRASION RESISTANCE ON POLYFUNCTIONAL REACTIVE DYED (H.E) LYOCELL

The results of this test should not be used indiscriminately particulars not for comparing fabrics of widely different fiber composition or construction. This present study for abrasion resistance of Alkali and Polyfunctional dyed (H.E method) lyocell fabrics was determined with a Martindale abrasion resistance tester according to ISO 12947-2- 1999 method. *For this present study,* both parameters such as concentration and type of alkalis are directly influenced the abrasion resistance of lyocell fabrics. When lyocell treated with sodium hydroxide is decrease the abrasion resistance than when we used for Tetra methyl-ammonium hydroxide for scouring. Effect of abrasion resistance on alkali treatments of lyocell is respectively *Sodium hydroxide < Potassium hydroxide < Lithium hydroxide < Tetra methyl- ammonium hydroxide < Polyfunctional reactive dyes (high exhaustion method),* When we dyed those alkali treated fabric with Polyfunctional reactive dyes (high exhaustion method) it will increase the abrasion resistance up to 28800 rubs per end point, whereas the original fabric have 27300 rubs per endpoint, basically the Polyfunctional reactive dyes form a covalent bond with all the hydroxyl group of lyocell, this is the reason for improve the abrasion resistance. The test result were given in the bellow table no 4.5

Table 4.5 Abrasion Resistance test results of on Polyfunctional Reactive dyed (H.E) lyocell

S. No	H.E Method	
	Sample code	Abrasion Resistance [illegible] (End Point [illegible])
1.	Original	27300
2.	B1	26400
3.	B2	26800
4.	B3	27600
5.	B4	27300
6.	B5	28400
7.	B6	28650
8.	B7	28500
9.	B8	28700
10.	B9	29100
11.	B10	28800
12.	B11	29600
13.	B12	30000

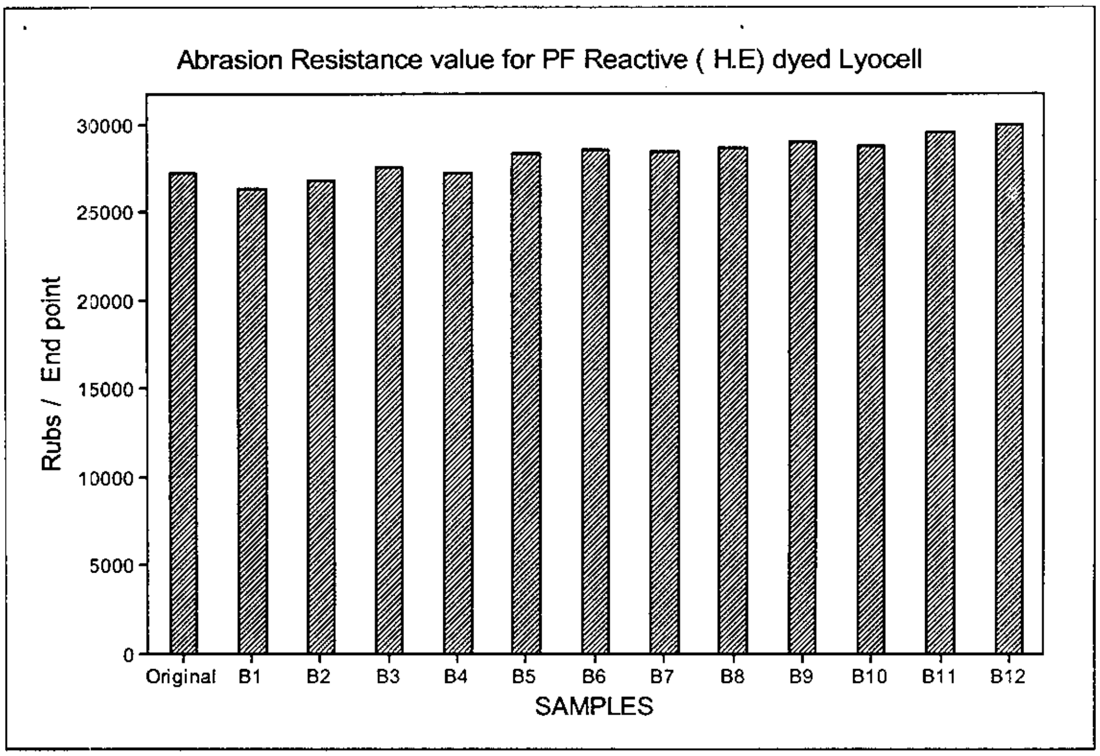


Figure 4.5 The Bar chart representation of the Abrasion resistance test on Polyfunctional Reactive dyed lyocell

4.6 EFFECT OF ABRASION RESISTANCE ON POLYFUNCTIONAL REACTIVE DYED (MIGRATION) LYOCELL

This present study for abrasion resistance of Alkali and Polyfunctional dyed (H.E method) lyocell fabrics was determined with a Martindale abrasion resistance tester according to ISO 12947-2- 1999 method. *For this present study,* both parameters such as concentration and type of alkalis are directly influenced the abrasion resistance of lyocell fabrics. When lyocell treated with sodium hydroxide is decrease the abrasion resistance than when we used for Tetra methyl- ammonium hydroxide for scouring. Effect of abrasion resistance on alkalis is respectively *Sodium hydroxide < Potassium hydroxide < Lithium hydroxide < Tetra methyl- ammonium hydroxide*, When we dyed those alkali treated fabric with Polyfunctional reactive dyes (high exhaustion method) it will increase the abrasion resistance up to 30300 rubs per end point, whereas the original fabric have 27300 rubs per endpoint, basically the migration method of dyeing increase the dyeability due to the higher movement of reactive dyes, due to the dyeability increasing the covalent bond between hydroxyl group of lyocell and cholorotriaznyl group of Polyfunctional reactive dyes, it caused to increase the abrasion resistance value of lyocell fabrics. The test result were given in the bellow table no 4.6,

Table 4.6 Abrasion Resistance test results of on dyed (Migration) lyocell

S. No	Migration Method	
	Sample code	Abrasion Resistance [illegible] Kpa (End Point Found @ Rubs)
1.	Original	27300
2.	C1	26900
3.	C2	27100
4.	C3	27500
5.	C4	28000
6.	C5	28300
7.	C6	28800
8.	C7	28400
9.	C8	28900
10.	C9	29400
11.	C10	29300
12.	C11	29900
13.	C12	30300

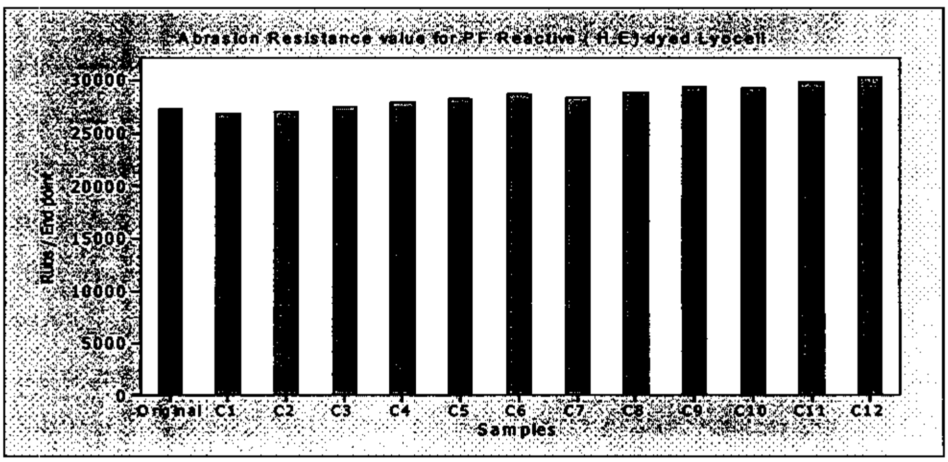


Figure 4.6 The Bar chart representation of the Abrasion resistance test on Polyfunctional Reactive dyed lyocell

4.7 SEM ANALYSIS ON ALKALI TREATED LYOCELL

In order to clarify the structural change during alkaline treatments, image analysis was performed using ***SEM***. Figure 4.8 gives the images of surface structure of fiber treated in NaOH solution. The large bundles or layers of macro fibrils are clearly observed on the surface of the fibers treated with different alkali solutions. The fiber treated with 1 gpl of Tetra methyl- ammonium hydroxide shows a smooth surface without any bundle and layer of macro fibrils. The fiber surface treated with 1 gpl of NaOH is less rough than that treated with 3 gpl of NaOH. The less fibrillation was observed on the fiber with the smooth surface without the bundle or the layers of macro fibrils. The formation of the bundle and the layers of the macro fibrils clearly elevate the fibrillation.

These results suggest that the treatments with alkalis cause the re-orientation of fibril structure and the fibrillation tendency is related to re-orientate micro fibril structure. When the macro fibrils are re-orientated unequally, the bundles or the layers of the macro fibrils generate and the fiber shows high fibrillation tendency. Contrarily, the fibrillation is retarded if the uniform re-orientation of fibrils is induced by the treatments. In this study, the uniform reorganization of the macro fibrils was obtained by the alkaline treatments with NaOH, KOH and Li OH. Generally the fiber treated with 1 gpl of Tetra methyl- ammonium hydroxide shows a smooth surface without any bundle and layer of macro fibrils and fibrillation structure that result shown in the figure 4.11. The less fibrillation was observed on the fiber with the smooth surface without the bundle or the layers of macro fibrils. The formation of the bundle and the layers of the macro fibrils clearly elevate the fibrillation. Also we get less fibrillation when we are treating with Lithium Hydroxide too, the result shown in the figure 4.10.

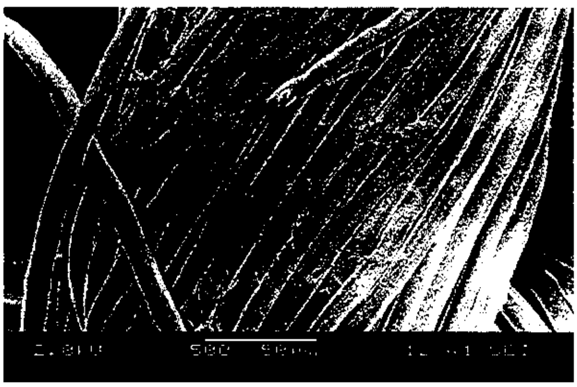


Figure 4.7 Scanning Electron Microscopic picture of original Lyocell fiber

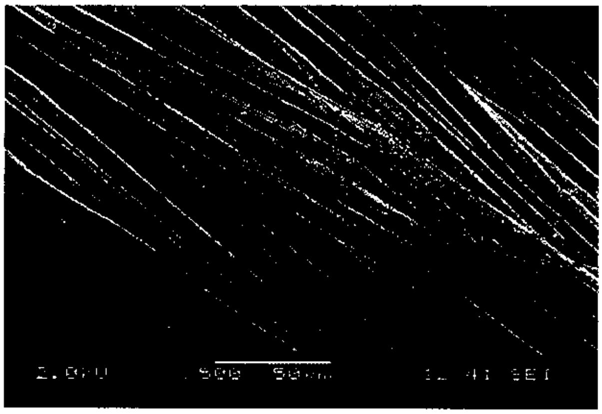


Figure 4.8 Scanning Electron Microscopic picture of Sodium hydroxide treated Lyocell fiber

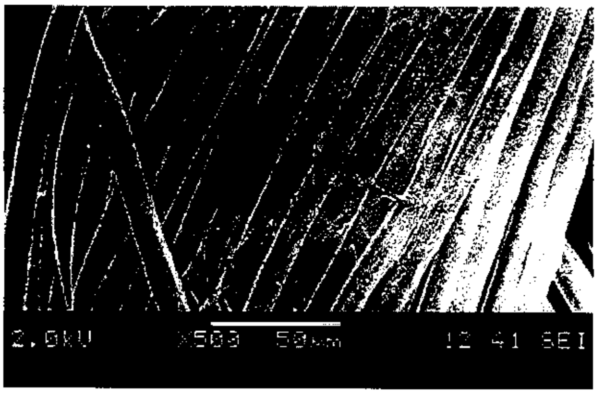


Figure 4.9 Scanning Electron Microscopic picture of Potassium hydroxide treated Lyocell fiber

Figure 4.10 Scanning Electron Microscopic picture of Lithium hydroxide treated Lyocell fiber

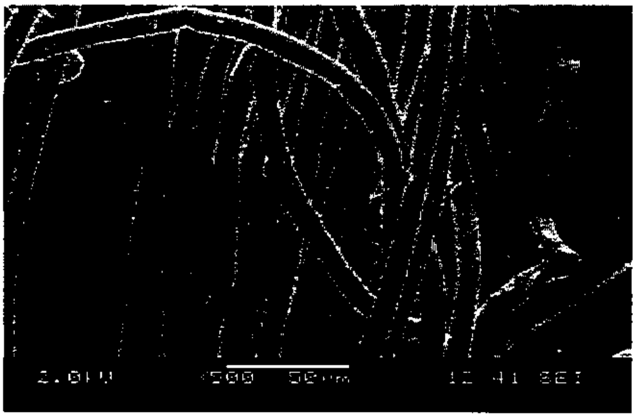


Figure 4.11 Scanning Electron Microscopic picture of Tetra methyl- ammonium hydroxide treated Lyocell fiber

4.8 SEM ANALYSIS ON DYED (HIGH EXHAUSTION & MIGRATION) LYOCELL

This study was initiated to observe the impact of fibrillation properties of Polyfunctional reactive dyeing process on the surface characteristics of the lyocell fabrics. The reaction of fabrics due to laundering (wet treatment) and dry cleaning as well as their appearance after these treatments. Taking this situation into consideration, the aim was to study the variation surface characteristic of Polyfunctional reactive dyed and followed by several laundered the fabrics. The formation of fibrillation and the contribution of fibrils to pilling process during the production and cleaning stages were investigated in detail, and Scanning Electron Microscopic was used to study the various stages of fibrillation were treated with different alkali's & Polyfunctional reactive dyed fabrics.

Generally the fiber treated with 1 gpl of Tetra methyl- ammonium hydroxide and the same samples dyed with Polyfunctional reactive dyes (migration method) shows a smooth surface without any bundle, layer of macro fibrils and fibrillation structure that result shown in the figure 4.13 and 4.12 show the result of 1 gpl of Tetra methyl- ammonium hydroxide and the same samples dyed with Polyfunctional reactive dyes (High Exhaustion method) Here too less fibrillation was observed on the fiber with the smooth surface without the bundle or the layers of macro fibrils. The formation of the bundle and the layers of the macro fibrils clearly elevate the fibrillation.

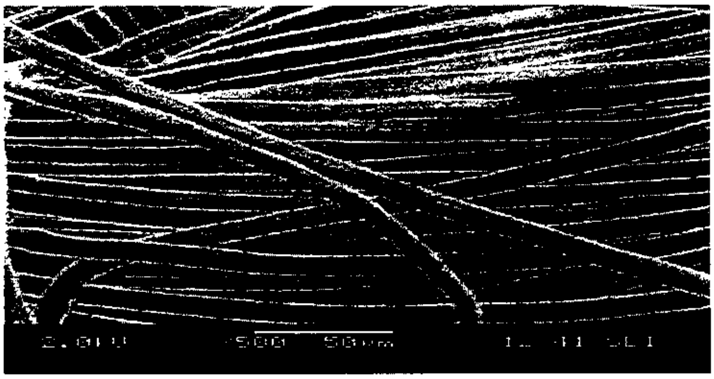


Figure 4.12 Scanning Electron Microscopic picture of Alkali treated and PF Reactive Dyed (H.E) Lyocell fiber

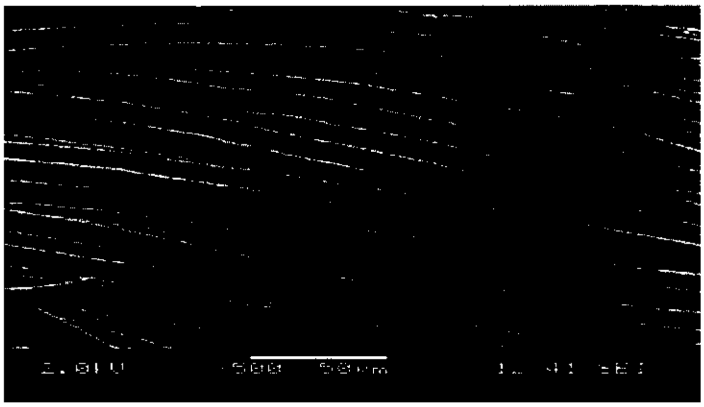


Figure 4.13 Scanning Electron Microscopic picture of Alkali treated and PF Reactive Dyed Lyocell fiber

4.9 EFFECT OF COLOR STRENGTH ON DYED LYOCELL (AATCC 182: 2005)

Table 4.7 Color Strength test results of Alkali + Polyfunctional Reactive dyed (H.E & Migration) lyocell

S.no	Alkali Concentration	K/S Value	Alkali Concentration	K/S Value
1.	Conventional Reactive dyed Lyocell 1 % Shade	10.7	Conventional Reactive dyed Lyocell 1 % Shade	10.7
2.	Na OH/1/ H.E	11.2	Na OH/1/ Migration	11.4
3.	Na OH/2 /H.E	11.6	Na OH/2 /Migration	11.8
4.	Na OH/3 /H.E	11.9	Na OH/3 /Migration	12.3
5.	K OH/1 /H.E	11.2	K OH/1 /Migration	11.3
6.	K OH/1.5 /H.E	12.0	K OH/1.5 /Migration	12.1
7.	K OH/2 /H.E	12.3	K OH/2 /Migration	12.8
8.	Li OH/0.5 /H.E	11.6	Li OH/0.5 /Migration	11.9
9.	Li OH/1 /H.E	12.3	Li OH/1 /Migration	12.7
10.	Li OH/1.5 /H.E	12.9	Li OH/1.5 /Migration	13.4
11.	Ta OH /0.25 /H.E	11.9	Ta OH /0.25 /Migration	12.4
12.	Ta OH /0.5 /H.E	12.7	Ta OH /0.5 /Migration	13.2
13.	Ta OH /1 /H.E	13.0	Ta OH /1 /Migration	13.9

From the table following observations are made.

- The test results were analyzed with the standard of AATCC 182: 2005
- K/S value Conventional dyed sample is comparatively lower than the K/S value of Polyfunctional Reactive dyed lyocell.
- K/S value is found to be more than High Exhaustion method when same concentration used in migration method.
- Maximum K/S value found at combination of 1 gpl of Tetra methyl- ammonium hydroxide and followed by Polyfunctional reactive (Migration) dyeing.

4.10 EFFECT OF WASHING AND RUBBING FASTNESS ON POLYFUNCTIONAL REACTIVE DYED LYOCELL (Both Method, H.E & Migration)

During wet treatments, such as washing treatment the adjacent undyed materials may take up color due to the transfer of dye from the original dyed material. This is known as staining. Basically for fastness property of dye depends upon the chemical structure of the colorant, dye concentration, environmental condition, nature of fiber, atmospheric contaminants, presence of foreign substances and state of dye inside the fabric.

Crocking relates to the property by which a textile transfers a colorant(s) from the surface of a colored yarn or fabric to another surface or adjacent area of the same fabric principally by rubbing. A wet crocking benefit is defined as fabric crocking rating greater than 3 after 1 launderings, preferably greater than 3 after 10 launderings, more preferably a greater than 3 after 25 washings. This present test should be conducted as per the AATCC test method of 8 & 107: 2002. The fastness properties of Polyfunctional reactive dyed fabric were given the table 4.8 and 4.9

Table 4.8 Color and Rubbing Fastness test results of Alkali + Polyfunctional Reactive dyed (H.E) lyocell

Sample No.	Sample code	Colour fastness to washing		Colour fastness to rubbing	
		Shade change	Staining on cotton	Dry rub	Wet rub
1	B1	4	4	3	3-4
2	B2	4	4	3-4	3-4
3	B3	4	4	4	3-4
4	B4	4	4	3-4	4
5	B5	4	4	4	3-4
6	B6	4	4	4	4
7	B7	3-4	3	4	3-4
8	B8	4	3-4	3-4	3-4
9	B9	3-4	3	4	3-4
10	B10	4	4	4	4
11	B11	4	4	4	4
12	B12	4	4	4	4

Table 4.9 Color and Rubbing Fastness test results of Alkali + Polyfunctional Reactive dyed (Migration) lyocell

Sample No.	Sample code	Colour fastness to washing		Colour fastness to rubbing	
		Shade change	staining on cotton	Dry rub	Wet rub
1.	C1	3-4	4	4	3-4
2.	C2	4	4	4	4
3.	C3	4	4	4-5	4
4.	C4	3-4	4	4	4
5.	C5	4	4	4	4
6.	C6	4	3-4	4-5	4-5
7.	C7	3-4	3-4	4	3
8.	C8	4	3-4	4	3
9.	C9	4	4	4-5	4
10.	C10	4	4	4	4
11.	C11	4	4	4-5	4
12.	C12	4-5	4	4-5	4-5

4.11 EFECT OF LIGHT FASTNESS ON POLYFUNCTIONAL REACTIVE DYED LYOCELL

This test measures the resistance to fading of dyed textiles when exposed to daylight. The test result were shown in the table 4.10, the Polyfunctional Reactive dyes have excellent light fastness properties, due to the more number of functional group present in the dye molecule, particularly lyocell were treated with 1 gpl of Tetra methyl- ammonium hydroxide and the same samples dyed with Polyfunctional reactive dyes (High Exhaustion and Migration method) showing outstanding light fastness properties, This present test should be conducted as per the AATCC test method of 16 : 2002, also the table 4.10 shows the fastness properties of migration method slightly higher than the high exhaustion method, due to the dyeing parameter.

Table 4.10 Light Fastness test results of Alkali + Dyed lyocell

S. No	H.D Method		Migration method	
	Sample code	Color Fastness to Light	Sample code	Color Fastness to Light
1.	B1	7	C1	6
2.	B2	7	C2	6-7
3.	B3	7	C3	6
4.	B4	7	C4	6-7
5.	B5	6-7	C5	7
6.	B6	6	C6	7
7.	B7	7	C7	6-7
8.	B8	7	C8	6
9.	B9	7	C9	6-7
10.	B10	7	C10	6
11.	B11	7	C11	6
12.	B12	7-8	C12	7-8

CHAFTER- 5

CONCLUSIONS

From the study conducted on the effect of alkali and dyeing treatment on fibrillation properties of lyocell fiber, the following conclusions are derived.

- Treating the lyocell with alkali's which will form a Na-cellulose II, during this process the lyocell undergo plasticization. Which in turn reduce the fibrillation tendency of the lyocell. Particularly when we treated the lyocell with 1 gpl of Tm AH, the Na- Cellulose II formation is higher; it causes to reduce the fibrillation tendency compare to sodium Hydroxide.

- Reactive dyeing processes with Polyfunctional dyestuffs can crosslink the fiber and thereby prevent or inhibit the fibrillation of the lyocell.

- The scope of this present work was to investigate changes on the surface of lyocell fibers due to fibrillation formation. The pilling, abrasion properties and fibrillation properties can be investigated in this work.

- The fibrillation formation of fabrics after both treatments (alkali and PF reactive dyes) is reduced than above single treatment. Compare to conventional method it reduce the fibrillation tendency of lyocell fabrics.

- The mechanism of pill formation including fibrillation process was proposed taking into account the effects of consecutive wash and dry treatments. Normally fibers came out from yarn by mechanical abrasion due to low fiber/ fiber friction; they are fibrillated and tangled owing to the softness and high fiber/ fiber friction in wet condition.

- Pilling resistance can be higher when we dyed lyocell with Polyfunctional reactive dyes particularly migration method, the same method will help to increase the Abrasion resistance too.

5.1 RECOMMENDATIONS FOR FUTURE WORK

The present investigations have paved the way for further research on the following aspects;

- The studies can be extended to application of Environmental friendly Cross linking agent for lyocell and dyed with PF reactive dyes.

- The studies can be extended to apply the advance enzymatic treatment to lyocell; currently the cellulase enzyme cannot give good result against on fibrillation and other related properties.

- The studies can be extended to application of Environmental friendly Cross linking agent through Nano technology for lyocell and dyed with PF reactive dyes.

- Further study can be done to produce commercially viable Tm AH.

CHAFTER – 6

LIST OF PUBLICATIONS

1. Aravin Prince Periyasamy , Dr (Mrs) Bharathi Dhurai., Dr K.Thangamani - A Study on Fibrillation Properties of Lyocell Fiber- Colourage- India to be published in Jan-2011.

2. Aravin Prince Periyasamy , Dr (Mrs) Bharathi Dhurai., Dr K.Thangamani - *An Overview of Processing and Application of Lyocell-* Textile Review- India to be published in Jan-2011.

3. Aravin Prince Periyasamy , Dr (Mrs) Bharathi Dhurai - *A New Method of Dyeing of Lyocell / Cotton Fabrics With Reactive Dyes* – Accepted for Publication in the Journal of Cotton Science - USA on 19 Jan-2011.Letter Receipt JCS 08-055.pdf

4. Aravin Prince Periyasamy , Dr (Mrs) Bharathi Dhurai - *A New Method of Dyeing of Lyocell With Poly functional Reactive Dyes* – Accepted for Publication in the Pakistan Textile Journal - Pakistan on 21 Feb 2011.

5. Aravin Prince Periyasamy , Dr (Mrs) Bharathi Dhurai - *Effect of Fibrillation on Pilling Tendency of Lyocell Fiber–* Accepted for Publication in the Indian Textile Journal - India on 08-Mar-2011.

CHAPTER 7

REFERENCE

1. AATCC Standard test method (107-2002), 'Determination of color fastness to washing.
2. AATCC Standard test method (8-2005), 'Determination of color fastness to crocking.
3. Bates I, Mauchru E, Phillips DAS, Renfrew AHM, Su Y, Xu J (2004) Cross-linking agents for the protection of lyocell against fibrillation: synthesis, application and technical assessment of 2,4-diacrylamidobenzenesulphonic acid. Color Technology 120:293–300. doi:10.1111/j.1478-4408.2004.tb00233.
4. Brauneis F. and Eibl M. 1998. Finishing of knit goods produced from Lenzing Lyocell. Melliand Textile 79(3): 155-156.
5. Bochek AM, Effect of Hydrogen Bonding on Cellulose Solubility in Aqueous and Nonaqueous Solvents, (2003) Russian Journal of Applied Chemistry, Vol. 76(11), 1711-1719
6. Bredereck K., Stefani H.-W., Beringer J. and Schulz F. 2003. Alkali- und Fluessigammoniak-Behandlung von Lyocell fasern. Melliand Textile- 12:58-65
7. Blackburn (2005).-Biodegradable and sustainable fibers- Woodhead Publishing Limited and CRC Press LLC ©
8. Chavan RB, Patra AK (2004) Development and processing of lyocell. Indian Journal Fiber & Textile Research – vol 29:483–492.
9. Chae D. W., Choi K. R. and Kim B. C. (2003). Effect of cellulose pulp type on the mercerizing behavior and physical properties of lyocell fibers. Textile Research Journal 73(6): 541-545.12.
10. Chae DW, Chae HG, Kim BC, Oh YS, Jo SM, Lee WS (2002) Physical properties of lyocell fibers spun from isotropic cellulose dope in NMMO monohydrate. Textile Research Journal 72:335–340. doi:10.1177/004051750207200410
11. Colom X, Carrillo F (2002) Crystallinity changes in lyocell and viscose-type fibers by caustic treatment. Europe Polymer Journal 38:2225–2230. doi:10.1016/S0014-3057(02)00132-5
12. Compounds useful for the preparation of bi-and poly-functional reactive dyestuffs United States Patent (2006) 5686642

13. Fink. H.-P., Weigel P., Purz H. J. and Ganster J. 2001. Structure formation of regenerated cellulose materials from NMMO-solutions-. Polymer Science- 26: 1473
14. Gandhi et. al., (2002) "A Novel Route for Obtaining 'Peach Skin Effect' on Lyocell and its Blends", AATCC Review, No. 4 page 48-52
15. Georgieva, D.Pishev 2001-Dyeing Of Cellulose Textile Materials With Mono - And Polyfunctional Reactive Dyes- Journal of the University of Chemical Technology and Metallurgy, XXXVI, Book 2, - Sofia, Bulgaria.
16. Goswami P, Blackburn RS, Taylor J, Westland S, White P (2007) Dyeing behavior of lyocell fabric effect fibrillation. Color Technology 123:387–393
17. Goswami P, Blackburn RS, El-Dessouky HM, Taylor J, White P (2009) Effects of sodium hydroxide pre-treatment on the optical and structural properties of lyocell. Europe Polymère Journal 45:455–465. doi:10.1016/j.eurpolymj.2008.10.030
18. Henrissat B., Marchessault RH et al., (1987) A Solid-State C-13 NMR Study of the Cellulose IEthylenediamine Complex, Polymer Communications, Vol. 28(4), 113-115
19. Hunter M Renfrew- 1999 Reactive Dyes for Textile Fibers: The chemistry of activated p-bonds as reactive groups and miscellaneous topics Society of Dyers and Colorists.
20. Horii F, Hirai A et al., (1983) Solid-State C-13 NMR Study of Conformation of Oligosaccharides and Cellulose – Conformation of CH2OH Group about the Exo-Cyclic C-C Bond, Polymer Bulletin., Vol. 10(7-8), 357-361
21. Ibbett R. N., and Hsieh Y. L. 2001. Effect of Fiber Swelling on the Structure of Lyocell Fabrics. Textile research journal 71(2): 164-173.
22. Isogai A, Usuda M et al., (1989) Solid-State CP/MAS C-13 NMR Study of Cellulose Polymer , Macromolecules, Vol. 22(7), 3168-3172
23. ISO Standard Test method for 12945-1-1998; Determination of Pilling resistance of textile fabrics.
24. ISO Standard Test method for 12949-1-1999; Determination of Abrasion resistance of textile fabrics.
25. Joonseok Koh- 2005 Dyeing Properties of a Mixed Bi-Functional Reactive Dye on a Novel Regenerated Cellulosic Fiber- IJFTR March Pp 88-93
26. Jakob B. and E. Agster, (1998). "Pretreatment and Finishing of Lyocell Woven Fabrics", International Textile Bulletin, No. 3, page 18-26.

27. J. M. Taylor, M. J. Bradbury and S. Moorhouse, (2001). "Dyeing Tencel and Tencel A100 with Poly-Functional Reactive Dyes", AATCC Review, No. 10 page 21-24.

28. J. M. Taylor and A. L. Harnden, 1997 "An Introduction to Tencel Processing", International Dyer, August, page 14.

29. Kasahara Katsuji et al., (2003). -The Effect of Reactive Dyeing and a Variety of Processing on the Fibrillation of Lyocell Fiber-Journal of the Japan Research Association for Textile-Vol.44; No.8; Page.480-486.

30. Laszkiewicz, B., (1997) Manufacture of Cellulose Fibers without the Use of Carbon Disulfide, ACGM LODART, SA, Lodz, Poland

31. Kim, J. J. Pak, S. M. Jo, W. S. Lee, (2005) Dry Jet-Wet Spinning of Cellulose/NMethylmorpholine N-Oxide Hydrate Solutions and Physical Properties of Lyocell Fibers, Textile Research. Journal., Vol. 75(4), 331-341

32. Lenz J. Schurz J. and Wrentschur E. 1992. Comparative characterization of solvent spun cellulose and high wet modulus viscose fibers by their long periods. Carbohydrate Polymers 43: 307.

33. Liu, J. Cuculo, B. Smith, (1989) Coagulation Studies for Cellulose in the Amonia/Amonium Thiocyanate (NH_3/NH_4SCN) Direct Solvent System, Journal of Polymer Science - Polymer Physics., Vol. 27, 2493-2511

34. Maia, E., Peguy, A., and Perez, S., Acta Crystallogr., Sect. B: Struct. Crystallogr. (1981) Crystal. Chemistry., Vol. 37(10), 1858

35. M. Nicolai et al (1996) Textile Crosslinking Reactions to Reduce the Fibrillation Tendency of Lyocell Fibers- *Textile Research Journal September vol. 66 no. 9 575-580.*

36. Nicolai M., Nechwatal A. and Mieck K.- P. (1998). Modified fibrillation behavior of solvent-spun cellulose fibers by the reaction with reactive dyes. International. Chem. 256: 21-27.

37. Nechwatal A., Nicolai M. and Mieck, K.- P. (1996). Crosslinking reactions of spunwet NMMO fibers and their influence on fibrillability. Textile Chemist and Colorist 28 (5): 24-27.

38. M. Nicolai., Nechwatal, (2006). A Fibrillation tendency of cellulosic fibers. VII. Combined effects of treatments with an alkali, crosslinking agent, and reactive dye-Journal of Applied Polymer Science, Vol.100, No.2, 1176-1183,

39. Nemec H. (1994). Fibrillation of cellulosic materials – Can previous literature offer a solution Lenzinger Berichte 74: 69-72.
40. Numata Y, Kono H et al., (2003) Cross-Polarization/Magic Angle Spinning C-13 Nuclear Magnetic Resonance Study of Cellulose I-Ethylenediamine- Complex Carbohydrate Polymers Vol. 96(5), 461-466.
41. Okubayashi S., Griesser U. and Bechtold T. (2004). A kinetic study of moisture sorption and desorption on lyocell fibers. Carbohydrate Polymers 58(3): 293-299.
42. Okubayashi S. and Bechtold T (2004). A Pilling Mechanism of Man-Made fibers- *Textile Research Journal* 2004; 96; 543
43. Rosenau, T., Potthast, A., Sixta, H., and Kosma, P., (2001) Polymer. Science., Vol. 26, 1763, 86
44. W. Udomkichdecha et al. (2002) Relationships Between Fibrillation Behavior of Lyocell Fibers and Their Physical Properties- *Textile Research Journal*; 72; 939 http://trj.sagepub.com/cgi/content/abstract/72/11/939
45. Toth T., Borsa J., Reicher J., Sally P., Sajo I. And Tanczos I. et al. (2003). Mercerization of cotton with tetramethylammonium hydroxide. Textile Research. Journal. 73(3): 273-278.
46. Wangsun Zhang, Satoko Okubayashi And Thomas Bechtold (2003) -Modification Of Fibrillation By Textile Chemical Processing- Lenzinger Berichte, 82 58-63.
47. Yi-Jun Pan, Chien-Kuo Yen-(2008) The relative characteristics between absorption-desorption curves and dyeing affinities of pre-treated Lyocell fiber- Journal of Applied Polymer Science, Vol.142, No.2, 92-98.
48. Yi-Jun Pan, Chien-Kuo Yen- (2005) Cellulosic Fabrics – Effects of Fibrillation. Textile Res. Journal 75 (4), 288- 292.
49. Zhang W., Okubayashi S. and Bechtold T. (2005). Fibrillation tendency of cellulosic fibers – Part 3. Effects of alkali pretreatment of lyocell fiber. Carbohydrate Polymers 59(2): 173-179,
50. **http://www.tencel.com/process parameters.html2345**
51. **http://www.lenzing.com/lyocell/properties**
52. **http://www.lenzing.com/lyocell/production.20954.html**
53. **http://www.lenzing.com/lyocell/processparameters.543.pdf**
54. **http://en.wikipedia.org/wiki/Lyocell accessed on 14-06-10**
55. **http://www.fibersource.com**

APPENDIX-I

One-way ANOVA: Pilling Resistance versus Alkali Treatment

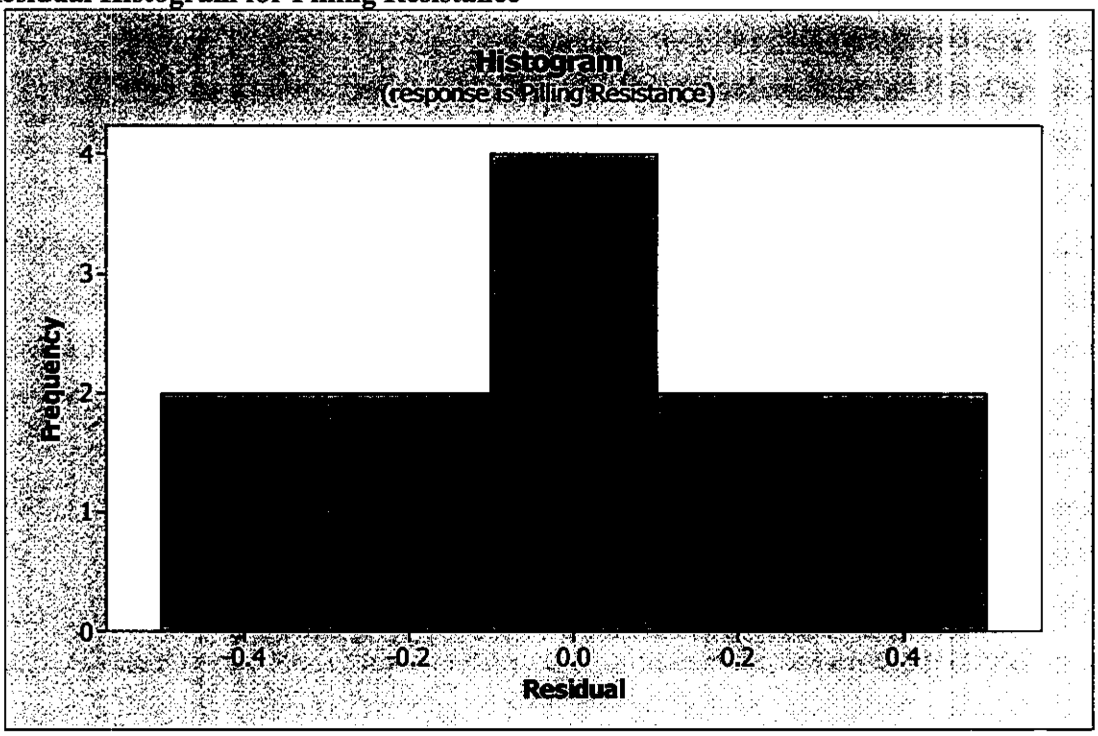
```
Source            DF     SS      MS     F      P
Alkali Treatment   3  1.229  0.410  3.93  0.054
Error              8  0.833  0.104
Total             11  2.063

S = 0.3227   R-Sq = 59.60%   R-Sq(adj) = 44.44%


                                 Individual 95% CIs For Mean Based on
                                 Pooled StDev
Level  N    Mean   StDev  ---------+---------+---------+---------+
1      3  3.0000  0.0000  (--------*--------)
2      3  3.1667  0.2887      (-------*--------)
3      3  3.5000  0.5000            (--------*--------)
4      3  3.8333  0.2887                   (--------*-------)
                          ---------+---------+---------+---------+
                                3.00      3.50      4.00      4.50

Pooled StDev = 0.3227
```

Residual Histogram for Pilling Resistance



INFERENCE

Table value (4.07), calculated value (3.93)

Calculated value is less than the Table value, ***so the difference is not significant.***

APPENDIX- II

One-way ANOVA: Pilling Resistance versus Alkali + PF Reactive dyed H.E

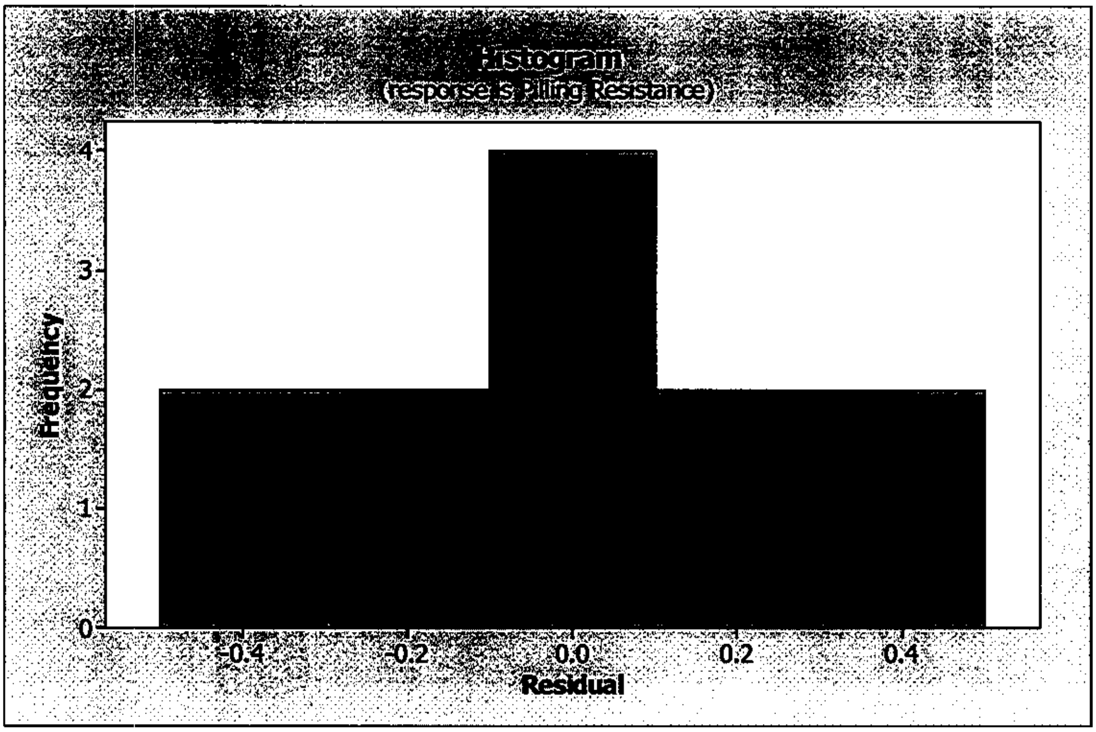
```
Source                   DF     SS     MS     F      P
Alkali + PF Reactive dye  3  1.750  0.583  4.67  0.036
Error                     8  1.000  0.125
Total                    11  2.750

S = 0.3536   R-Sq = 63.64%   R-Sq(adj) = 50.00%


                                Individual 95% CIs For Mean Based on
                                Pooled StDev
Level  N    Mean   StDev   ------+---------+---------+---------+---
1      3  3.1667  0.2887  (---------*---------)
2      3  3.6667  0.2887            (---------*---------)
3      3  4.0000  0.5000                  (---------*---------)
4      3  4.1667  0.2887                     (---------*---------)
                           ------+---------+---------+---------+---
                               3.00      3.50      4.00      4.50

Pooled StDev = 0.3536
```

Residual Histogram for Pilling Resistance



INFERENCE

Table value (4.07), calculated value (4.67)

Calculated value is more than the Table value, ***and then the difference is significant.***

APPENDIX – III

One-way ANOVA: Pilling Resistance versus Alkali and PF Reactive dyed (M)

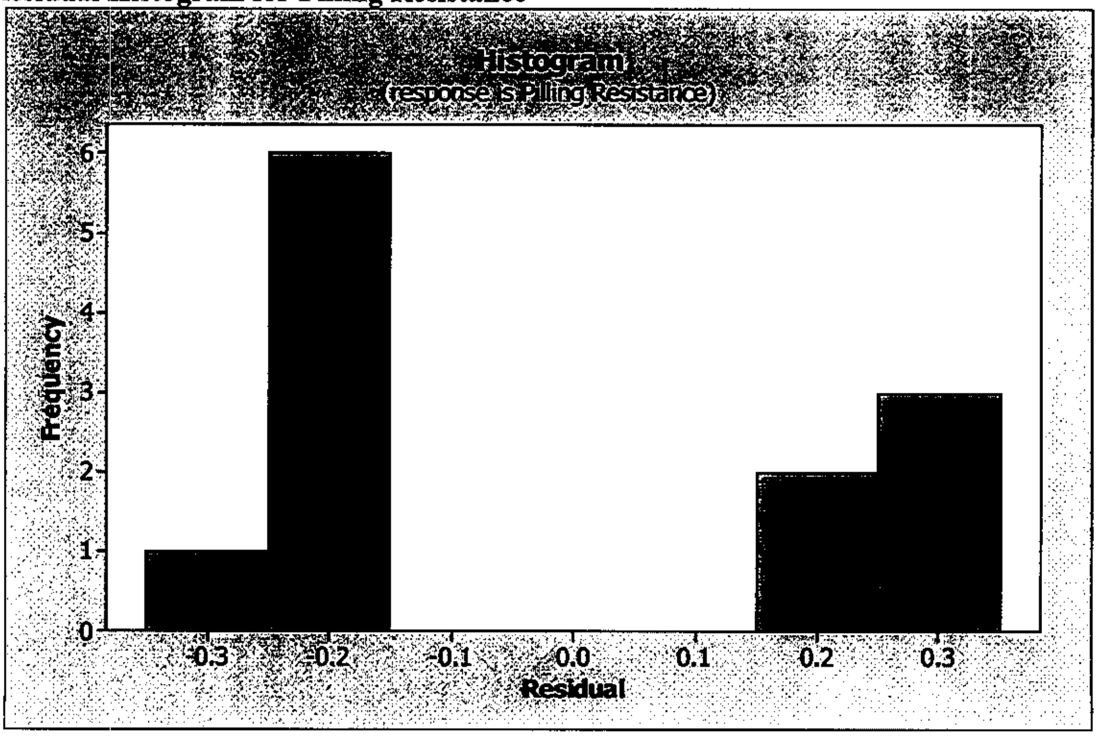
```
Source                    DF      SS      MS     F      P
Alkali and PF Reactive d   3  1.5625  0.5208  6.25  0.017
Error                      8  0.6667  0.0833
Total                     11  2.2292

S = 0.2887   R-Sq = 70.09%   R-Sq(adj) = 58.88%


                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level  N    Mean   StDev  ----+---------+---------+---------+-----
1      3  3.6667  0.2887  (------*-------)
2      3  4.1667  0.2887            (------*-------)
3      3  4.3333  0.2887               (-------*------)
4      3  4.6667  0.2887                      (------*-------)
                          ----+---------+---------+---------+-----
                            3.50      4.00      4.50      5.00

Pooled StDev = 0.2887
```

Residual Histogram for Pilling Resistance



INFERENCE

Table value (4.07), calculated value (6.25)

Calculated value is more than the Table value, ***and then the difference is significant.***

APPENDIX- IV

One-way ANOVA: Abrasion Resistance versus Alkali Treating

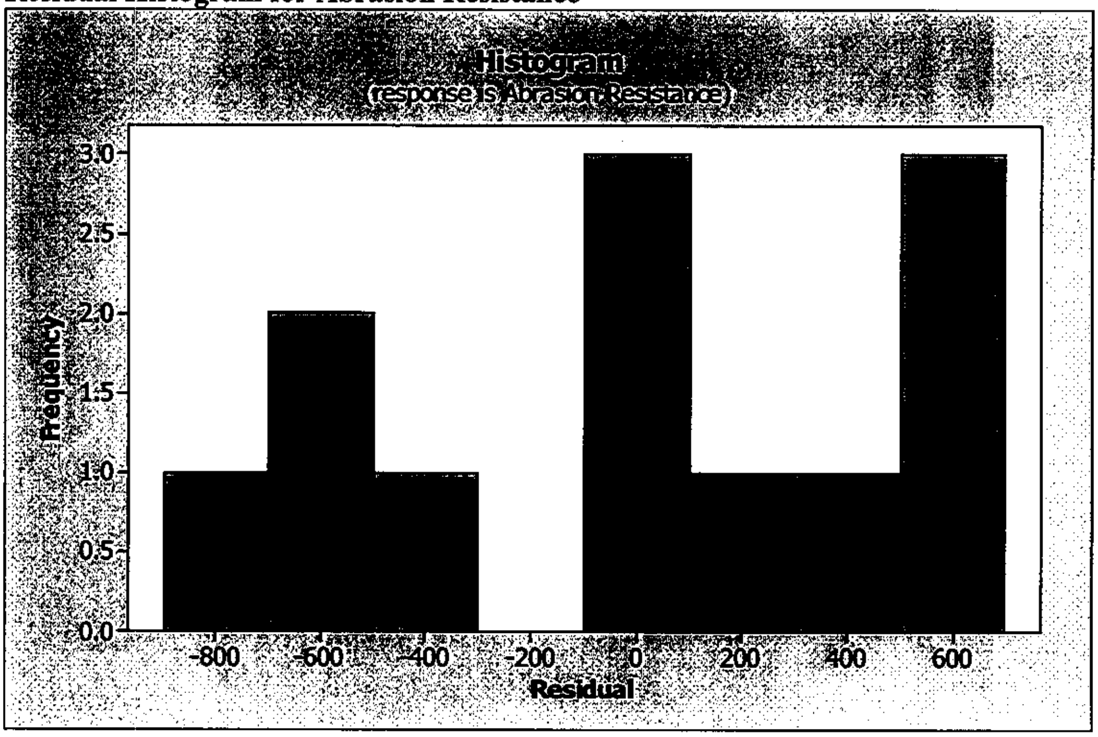
Source	DF	SS	MS	F	P
Alkali Treating	3	5249167	1749722	4.70	0.036
Error	8	2980000	372500		
Total	11	8229167			

S = 610.3   R-Sq = 63.79%   R-Sq(adj) = 50.21%

```
                                Individual 95% CIs For Mean Based on
                                Pooled StDev
Level  N   Mean  StDev  --------+---------+---------+---------+-
1      3  24967    603  (-------*-------)
2      3  25333    651     (-------*-------)
3      3  25800    458          (-------*-------)
4      3  26733    702                   (-------*-------)
                        --------+---------+---------+---------+-
                            25000     26000     27000     28000
```

Pooled StDev = 610

Residual Histogram for Abrasion Resistance



INFERENCE

Table value (4.07), calculated value (4.73)

Calculated value is more than the Table value, ***and then the difference is significant.***

APPENDIX- V

One-way ANOVA: Abrasion Resistance versus Alkali & PF Reactive Dyed (H.E)

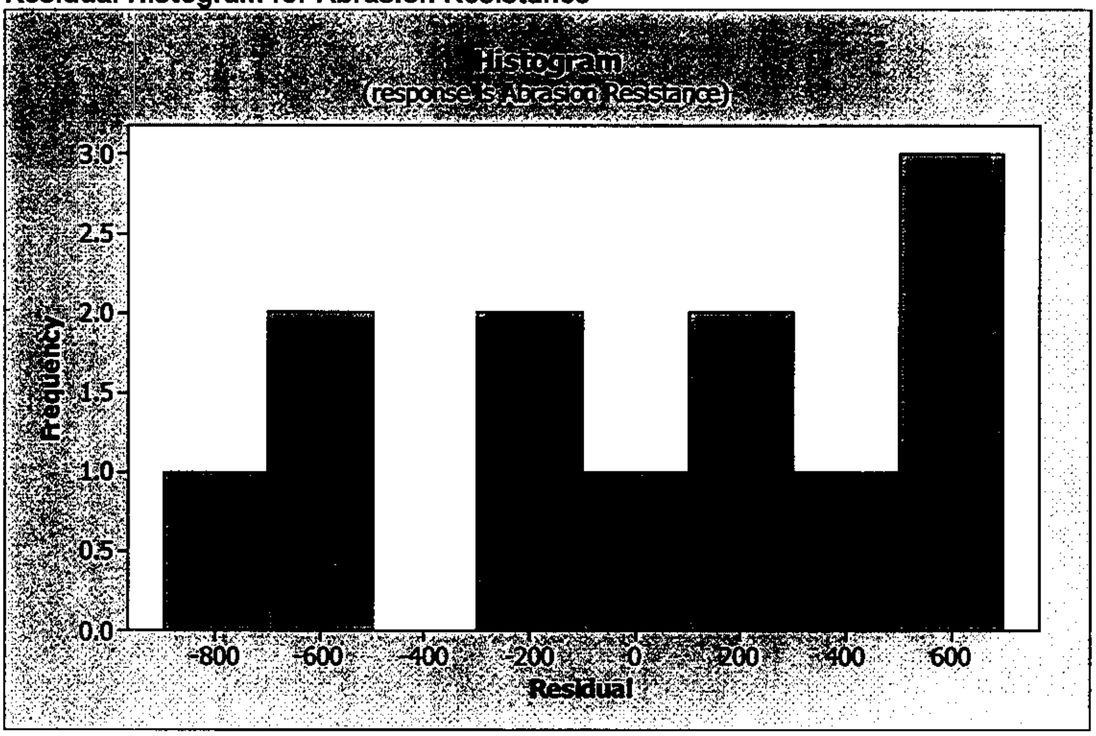
Source	DF	SS	MS	F	P
Alkali & PF Reactive Dye	3	10435625	3478542	10.26	0.004
Error	8	2711667	338958		
Total	11	13147292			

S = 582.2   R-Sq = 79.37%   R-Sq(adj) = 71.64%

```
                                Individual 95% CIs For Mean Based on Pooled StDev
Level  N   Mean  StDev     --+---------+---------+---------+-------
1      3  26933    611     (-----*------)
2      3  28117    718               (-----*------)
3      3  28767    306                    (------*-----)
4      3  29467    611                          (------*-----)
                           --+---------+---------+---------+-------
                         26400     27600     28800     30000
```

Pooled StDev = 582

Residual Histogram for Abrasion Resistance



INFERENCE

Table value (4.07), calculated value (10.26)

Calculated value is more than the Table value, ***and then the difference is significant.***

APPENDIX- VI

One-way ANOVA: Abrasion Resistance versus Alkali & PF Reactive (M)

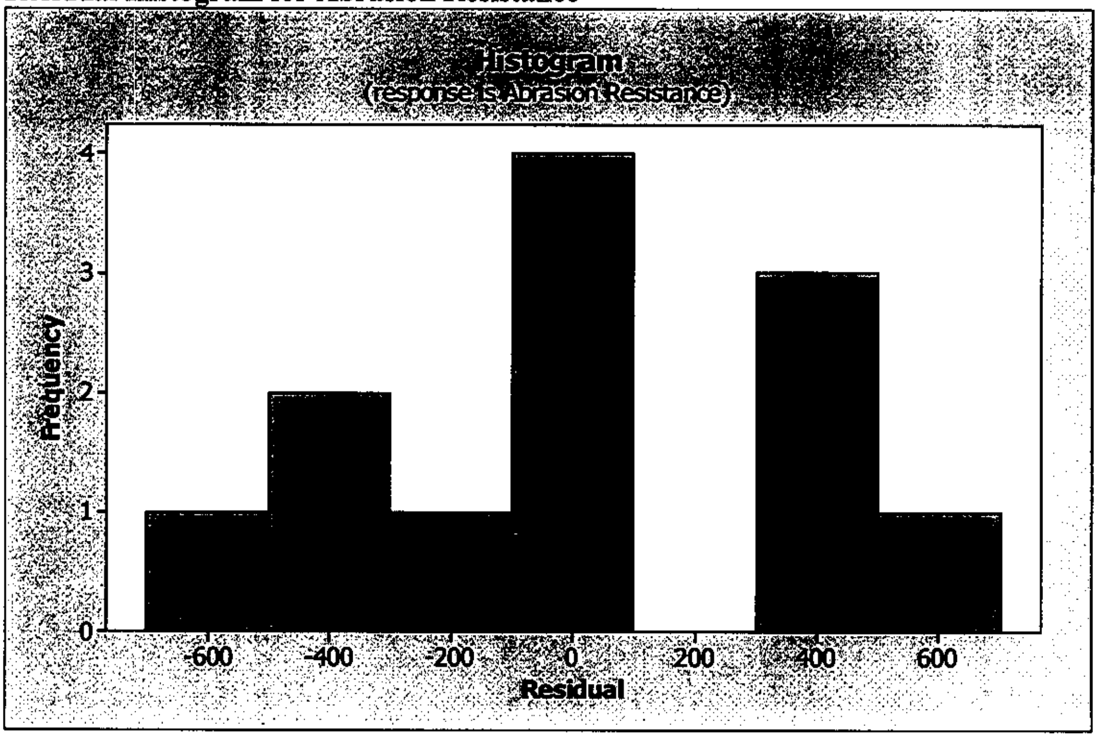
```
Source                   DF        SS       MS      F      P
Alkali & PF Reactive(M)   3  11146667  3715556  19.56  0.000
Error                     8   1520000   190000
Total                    11  12666667

S = 435.9   R-Sq = 88.00%   R-Sq(adj) = 83.50%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level  N   Mean  StDev  ----+---------+---------+---------+-----
1      3  27167    306  (-----*----)
2      3  28367    404             (-----*----)
3      3  28900    500                  (-----*-----)
4      3  29833    503                            (----*-----)
                        ----+---------+---------+---------+-----
                        27000     28000     29000     30000

Pooled StDev = 436
```

Residual Histogram for Abrasion Resistance



INFERENCE

Table value (4.07), calculated value (19.56)

Calculated value is more than the Table value, ***and then the difference is significant.***