CHAPTER 3

MATERIALS AND METHODS

Generally, different types of products are used for pretreatment of Cotton and Viscose rayon like Wetting agents, Enzymes, Salts, Alkalis, Acid, Bleaching agent, Stabilizers, Defoaming agents, chelating agents, etc. Out of these products, the present studies focus on modifying wetting agent and desizing agent. Several, formulations with different permutation & combinations have been studied and finalized Enzymatic desizing agent (EDA) & Polymeric wetting agent (PWA).

This project was divided in three phases. First study phase considered as making formulation and testing of desizing agent and wetting agent for cellulosic namely Cotton and Viscose rayon. The second study phase is application of that desizing and wetting agent on Cotton (woven & knitted) and Viscose rayon woven on lab scale to optimizing all parameters and developed optimistic process called as modifying pretreatment process. The third phase of study is purely industrial trial based where compared modified pretreatment process with current pretreatment process on Cotton and Viscose rayon.

3.1 PHASE–I: Eco-friendly pretreatment interventions for cotton and viscose rayon

3.1.1 Materials required to formulate Enzymatic desizing agent (EDA) & Polymeric wetting agent (PWA)

- **Polymer:** It is PET-PEG copolymer which is manufactured by Zydex industries. It is 100% solid brownish yellow product. It is Anionic in nature.
- **Emulsifier:** It is Non-ionic in nature of colorless transparent viscous liquid. It is mixture of lauryl and tridecyl alcohol.
- **Defoamer:** It is silicone based non-ionic nature defoamer. It is white flowable liquid.
- **Amylase Enzyme:** It is amylase based liquid enzyme. It is in yellowish brown in colour.

3.1.2 Machines used for formulation

Different machines like weighing balance, motor for homogenized mixing, electric heating mental are used for formulation of both the products.

3.1.2.1 Weighing Balance

Specification of weighing balance is as followed:

Manufacturer:	Essae -Teraoka Ltd
Serial No.:	G 85213434061
Electrical spec.:	230V, 13W, 50HZ
Capacity:	10gms – 3000 gms

3.1.2.2 Motor for homogenized mixing

Figure 3.1 Motor for Mixing with speed controller

Specification of Motor is as followed:

Manufacturer:	Remi elektrotechnik Ltd, Vasai
Type:	RQ – 134H
Electrical spec.:	2.4A, 230V, 50HZ, AC /DC
Capacity (RPM):	5000

3.1.2.3 Electric heating Mantle

Figure 3.2 Electric heating mantle

Manufacturer:	Scientific equipments
Electrical spec.:	300W, 230V

3.1.3 Experimental formulation method to formulate products EDA and PWA

3.1.3.1 Enzymatic desizing agent (EDA): Formulated product with Amylase Enzyme

Product description:

When starch is used as a sizing agent then one can go for the desizing by using Amylase type of enzyme. The general method of application of Amylase type of enzyme is by exhaust process. This EDA is unique blend for very good Wetting & Desizing action during desizing of 100% Viscose rayon, 100% cotton & their blended woven fabrics.

Formulation:

Different permutation and combination of EDA formulation were done in Zydex Lab. As mentioned in Table 3.1 was the final combination of EDA.

Table 3.1 Formulation component of EDA

Component	Concentration (%)
Polymer	21-25
Emulsifiers	50-54
Defoamer	0.5-2
Amylase Enzyme	24-28

Formulation guidelines:

Following procedure may be followed for formulating the product with emulsifier.

- This EDA was formulated product of Polyester-polyethylene glycol polymer, blend of emulsifier, Amylase Enzyme, defoamer and sequestering agent.
- Taken 100% Polyester –polyethylene glycol polymer in SS vessel.
- Melted this polymer at 110 ± 2 °C using electric heater.
- Added emulsifiers & defoamer simultaneously with slowly addition in the melted polymer under stirring.
- Keep stirring the mixture for around 30 minutes for homogenous mixing of the product.
- Cool it down to room temperature.
- Finally added amylase liquid enzyme in the above formulation and stirring at slow speed for another 5-10 mins.
- Product was ready to use.
- EDA was Viscous Flowable liquid product under ambient conditions (35 °C).

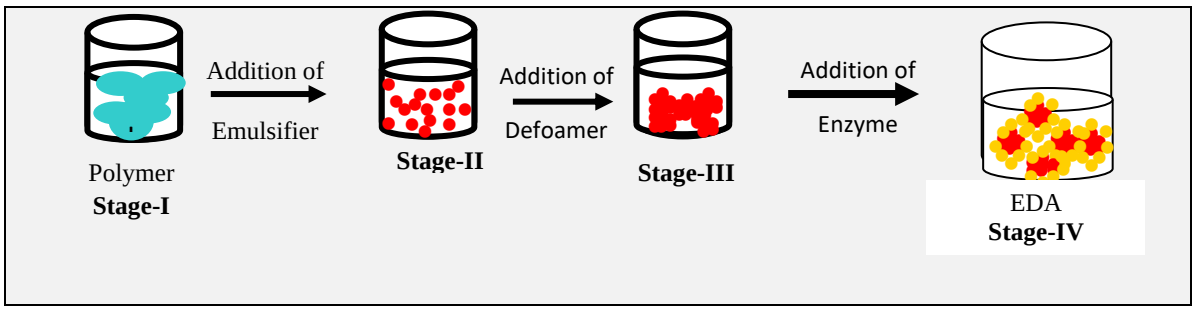

Figure 3.3 Schematic process diagram of formulation EDA

3.1.3.2 Polymeric Wetting agent/ cleaning agent (PWA)

Product description:

Unique blend of polymer and surfactants for very good re-wetting action during desizing, scouring, bleaching of 100% cotton, 100% polyester & their blended woven and knitted fabrics.

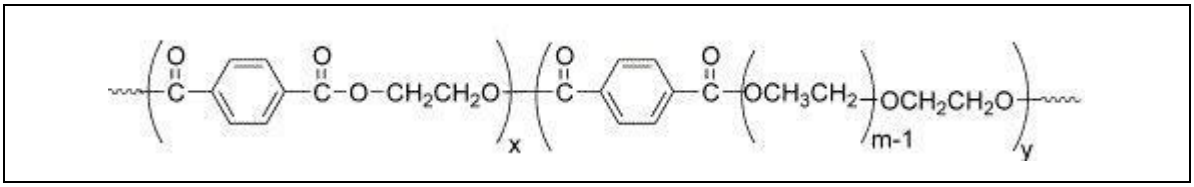

Figure 3.4 Structure of polymer

Formulation:

Different permutation and combination of PWA formulation were done in Zydex Lab. As mentioned in Table 3.2 was the final combination of PWA.

Table 3.2 Formulation component of PWA

Component	Concentration (%)
Polymer	28-32
Emulsifiers	66-70
Defoamer	1-4

Formulation guidelines:

Following procedure may be followed for formulating the product with emulsifier.

- This PWA was formulated product of Polyester-polyethylene glycol polymer, blend of emulsifier, defoamer and sequestering agent.
- Take 100% Polyester –polyethylene glycol polymer in SS vessel.
- Melted this polymer at 110 ± 2 °C using electric heater.
- Added emulsifiers & defoamer simultaneously with slowly addition in the melted polymer under stirring.
- Keep stirring the mixture for around 30 minutes for homogenous mixing of the product.
- Cool it down to room temperature.
- Product was ready to use.

- PWA was viscous flowable liquid product under ambient conditions (35°C).

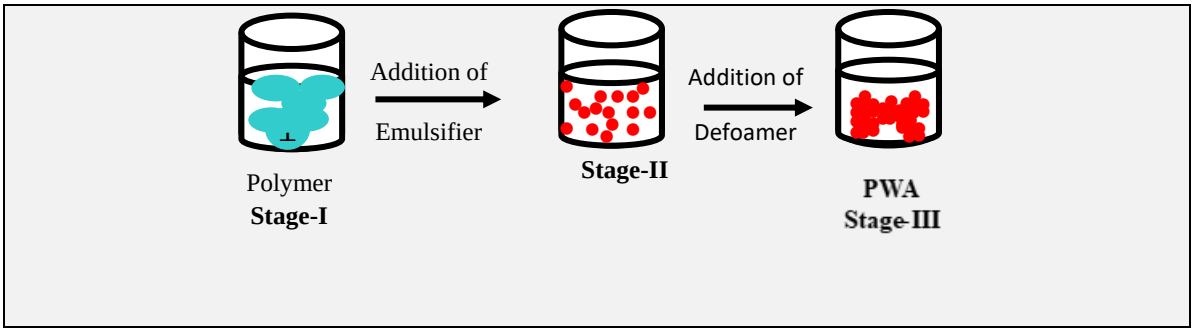

Figure 3.5 Schematic process diagram of formulation PWA

3.1.4 Testing procedure of EDA and PWA

3.1.4.1 Physical appearance of the products

Take 200 gms sample in 250 ml glass beaker and check its physical appearance. Visually see the colour of the product at ambient temperature 30 ± 2°C.

3.1.4.2 Checking pH of the products

pH of solution was measure with digital pH meter by using pH electrode (Ag/ $AgCl_2$/ 3MKCl as a reference). Before using the instrument, it was calibrated using two standard solution pH – 4 and pH – 7 which prepared by using buffer tablets. Calibration was checked daily and standard pH buffers were replaced after every week. The pH electrode was stored in distilled water after use.

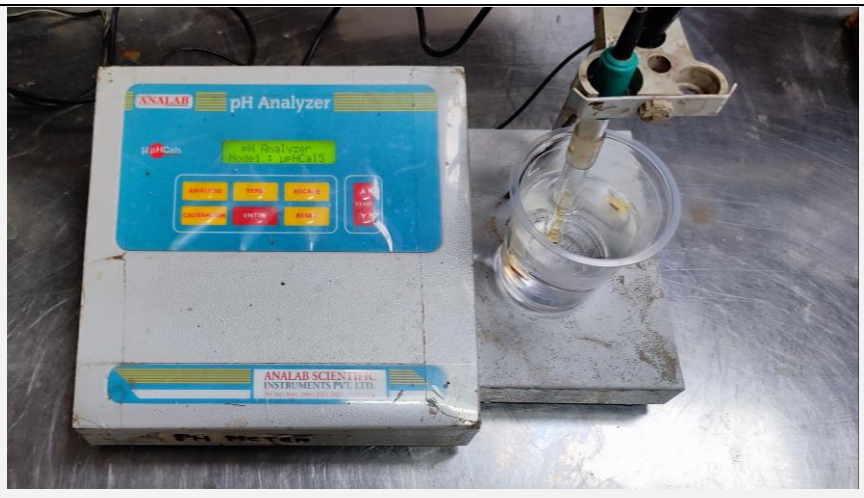

Figure 3.6 Analab scientific pH Analyzer

3.1.4.3 Viscosity of the products

- Checked viscosity of products in RVT model using spindle no 2 or 3 at 20 rpm at ambient temperature 30 ± 2°C.

- Rechecked viscosity after overnight ageing i.e. solution was kept at RT for overnight after covering it with thin plastic film or by keeping it in closed plastic bottle. There was tendency of increase in viscosity.
- Final clearance was given after checking overnight viscosity.

Figure 3.7 Brookfield viscometer RVT model

3.1.4.4 Specific gravity of the products

Specific gravity is checked with hydrometer at 28 to 32ºC. Put Hydrometer in prefilled product in 250 ml measuring cylinder and note reading.

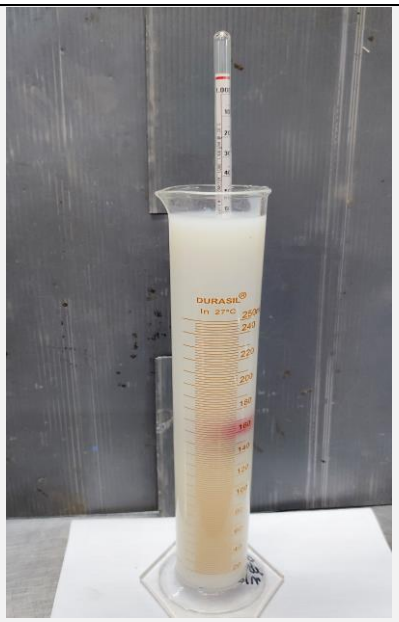

Figure 3.8 Hydrometer set up for specific gravity

3.1.4.5 Solid content of the products

Procedure to check solid content was as follows,

- Taken clean and dry petri-dish and weighted it. Let it be X g.
- Added approximately 1 - 1.10 gm of product and weighed it along with glass beaker. Let it be Y g.
- Keep petri-dish with product in oven at 110°C for 75 min.
- After 75 min taken out beaker from oven and allowed it to cool down to room temperature and then taken its weight. Let it be Z gm.
- Formula for Solid content was (Z-X / Y-X) x 100.

Figure 3.9 Petri-dish set up for solid content

3.1.4.6 Dispersibility of products in Hard and Soft water

Generally, all the products used in pretreatments were soluble in water due to transparent in nature. Here, I used polymer in both the products which seen as translucent nature in water and hence both the products are dispersible in water. Dispersibility of product is checked using borewell water of around 2000 TDS and RO water of around 20 TDS. Procedure was as follows:

- In 250 ml glass beaker, weighed 198gms of hard water/ soft water.
- Added 2 gms of product in water and mix it under manual stirring.
- It should get fully dispersed within 4-5 seconds and giving hazy solution.

3.1.4.7 Foaming test of the products

Foaming test was done by following steps:

- Prepared solution of 1% both the products in RO water.
- Taken 100 ml of 1% product in 250 ml of stoppered measuring cylinder.

- Done vigorous/ speedy shaking for 30 times.
- Opened stoppered from measuring cylinder and start stopwatch.
- Noted material height including foam in ml and time to taken settling of foam/ defoaming action time.

3.1.4.8 Stability of the products tested by AHS test

AHS is Accelerated Heat stability test to simulate stability of product during ageing. Random samples were kept under heat aging test at 60˚C for 14 days before giving clearance. The sample were kept under heat aging should not show any separation, flocculation or changed in viscosity.

3.1.4.9 Particle size distribution and Zeta potential

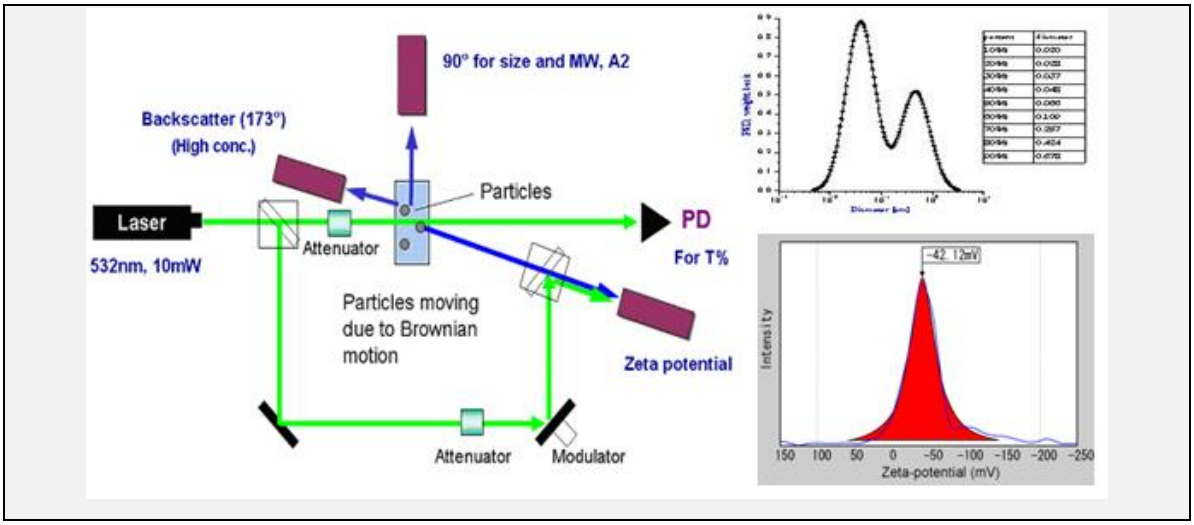

Figure 3.10 Zeta Particle Size Analyzer Schematic **(Bumiller)**

Sieve analysis, optical counting, electrical resistance counting, sedimentation, laser diffraction, dynamic light scattering, acoustic spectroscopy, and other methods can all be used to evaluate particle size and distribution (PSD). For determining size distribution, dynamic light scattering is the most popular technique.

Dynamic Light Scattering (DLS) Basic Principle

A well-known, non-invasive technique for figuring out the sizes of molecules and other tiny objects, typically in the submicron range and, with the most recent technology, down to 1 nanometer, is dynamic light scattering, also known as photon correlation spectroscopy (PCS) or quasi-elastic light scattering (QELS). Molecules, emulsions, and suspended particles all undergo Brownian motion. Solvent molecules, which are already moving due to their thermal energy, attack the area and create the motion. Smaller

particles are "kicked" further by the solvent molecules and move more quickly; as a result, when the molecules or particles are exposed to laser light, the intensity of the scattered light varies at a rate that is dependent on the size of the particles. Analysis of these intensity fluctuations yields the Brownian motion's velocity and, in turn, the particle size (radius rk) in accordance with the Stokes-Einstein relationship, which may be found at **(Weeks, 2007)**. The Stokes-Einstein relationship is illustrated in the following way:

$$r_k = \frac{kT}{6\pi\eta D}$$

Where, ***k*** - Boltzmann constant; ***D*** - Diffusion coefficient; ***η*** - Solvent viscosity; ***T***- Temperature in K

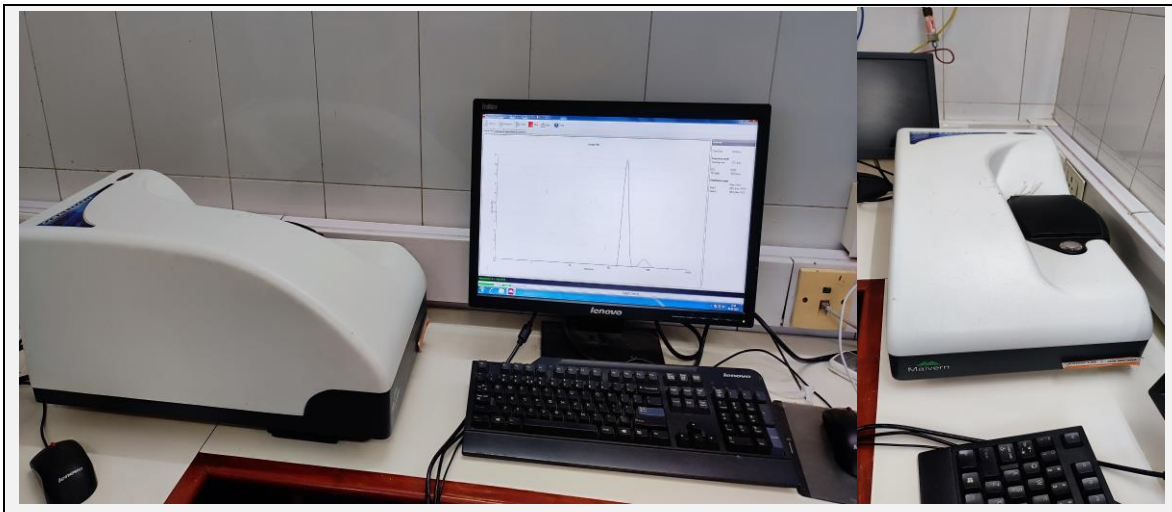

Figure 3.11 Malvern Particle Size Analyzer Set up

Applications

PWA & EDA were diluted 1000 times in beaker up till transparent solution form. Filled the diluted solution in cuvette. Clean the cuvette from outside by tissue paper. Put this cuvette in Particle size analyser and run the machine. Received results on display of attached computer screen. Textile substrates to get improved functionality by adding this type of textile chemicals. “The enhanced property is depended on the size of the applied product, which generally have a tendency to agglomerate. Therefore, size and size distribution study of the particle in the dispersion as well as suspension is important before applying to the textile substrates” as stated by Joshi, Bhattacharyya and Wazed in 2008 (Joshi, 2008). Measurements were done on Particle size analyzer with following specifications.

- Measurements: molecular size, molecular weight, particle size, and zeta potential
- Manufacturer: Malvern Panalytical Ltd, UK (Supplier: Aimil Ltd)
- Model Name: Zetasizer Nano ZS90 (Affordable Molecular/Particle Size and Zeta Potential Analyzer)
- Temperature range: 0°C to 90°C; Particle size range: 0.3nm to 5 µm; Static light scattering, electrophoretic light scattering, and dynamic light scattering technology
- Wet dispersion type; Cuvette is a sort of sample cell.
- Batch measurement for molecular measurements

3.1.4.10 Amylase activity in EDA

During the past decades much attention has been focused on the substitution of soluble enzyme-based analysis. This is because of the limited stability of the same enzymes under routine operating condition & there is relatively high loss of any analytical useful enzyme. Immobilized enzyme can be used continuously as compared to free enzyme. The successful utilization of immobilized enzyme in clinical chemistry may be derived into 3 categories i.e. enzyme reactor tube, analytical probes & dry reagent product.

Part A: To study the effect of pH on amylase activity.

Optional activity is often seen between pH values 5-7 when enzyme activity is evaluated at various pH levels. The pH activity shape at this range is determined as follows:

I) Enzyme denaturation at high or low pH.

II) Alteration of the enzyme or substrate's charge and state

III) By altering the substrate, the charge, or the decreased function related to substrate binding and catalysis, the pH can change the activity.

Consider a negatively charged enzyme to demonstrate this. With a positively charged substrate (SH+), this enzyme (-) responds.

Enzyme- + SH + Enzyme. SH

At low pH, negatively charged enzyme protonate & loose its negative charged.

Enzyme- + SH + Enzyme .H + S

At pH value, it lowers the effective concentration of enzyme- & SH+. Thus, lowering the reaction velocity when the enzyme & substrate are in the appropriate ionic stage. The maximum concentration of enzyme & substrate is correctly change at specific pH. Enzyme undergoes charges with varying pH, the charge group of enzymes which is still attached to the region where the substrate is bound, the charge on this will make protein more compact or dissociate into proteinomer or result in loss of activity.

Depending upon the severity, this changes their activity that may or may not restore when the enzyme become available to optimum pH.

Requirements:

1% starch, Enzyme, 0.1M phosphate buffer (pH–5.8.6.8,7.8), DNS reagent, spectrophotometer, Distilled water, test tubes, pipettes, boiling water bath, incubator.

Procedure:

- Take different aliquots of 1% starch solution & add 0.1M phosphate buffer to make volume 1ml having different pH.
- Add 1ml of enzyme in all the experimental tubes. (In blank add enzyme after incubation)
- For 15 min., incubate each the tubes at 37^0C.
- Add 1ml of DNS reagent to all tubes.
- Keep all the tubes in a boiling water bath for 10min & cool, and then add 9ml of D/W.
- Take O.D at 540nm.
- From the standard curve find the concentration of glucose “V”. Plot the graph of pH vs V.

Part B: To investigate how temperature affects the activity of enzymes.

The rate of an enzyme-catalyzed reaction slows down as the temperature rises, and this only holds true across a very small range of temperatures. The energy barrier for breaking the weak hydrogen bonds & hydrophobic interactions that preserve the 2º & 3º structures is first overcome by the increased kinetic energy of the enzyme, which causes the reaction rate to first increase as temperature rises. At this temperature, denaturation and precipitation lead to a decrease of catalytic activity, which is the major effect. The temperature of the cell in which an enzyme is found typically determines the temperature range throughout which it maintains a stable catalytically competent conformation. the

human enzyme that keeps the body temperature at 37°C. Generally, demonstrates stability between 45 and 55°C. Many of the microorganism-produced enzymes that prevent hot springs or extremely hot events in the ocean are stable at temperatures above 100°C. The optimum temperature of an enzyme is the temperature at which the greater amount of substrate gets changed in product per unit time.

Procedure:

- Take 1ml of standard starch solution & add 1ml of 0.1M phosphate buffer.
- Add 1ml of enzyme to all the experimental tubes. (After incubation add 1ml enzyme in all blank tubes)
- Incubate all the tubes for 15min at different temperature i.e., 0°C, 10°C, 37°C, 50°C.
- Add 1ml DNS reagent to all the test tubes.
- Keep the tubes for incubation & after that add 9ml D/W to all the tubes & measure O.D. at 540nm.
- From the standard curve, find out the concentration of sample.
- Plot the graph of T vs V.

Part C: To study the effect of incubation time on amylase activity.

A pH or temperature that is ideal cannot exist independently of time. Many enzymes from warm-blooded animals function best at a temperature of about 37°C. Hours, minutes, and days make up the unit of time. As a result, the time component plays a crucial role in defining other conditions that control the pace of enzyme action.

Procedure:

- Take 1ml of standard starch solution & add 1ml of 0.1M phosphate buffer.
- To all the experimental tubes, add 1ml of enzyme at 37°C.
- After incubation add 1ml of DNS reagent.
- Keep all the test tubes in boiling water bath for 10min.
- Cool the tubes & add 9ml D/W.
- Measure the O.D. at 540nm.
- From the standard curve find out concentration.
- Plot the graph time vs velocity.

3.2 Phase – II: Lab scale Pre-treatment of Cotton and Viscose rayon by modified process

The experimental strategy was centered on applying EDA and PWA via exhaust process to various fabrics. The samples of treated fabric were subsequently evaluated using a variety of methods. The effects of the treatment on the attributes were then assessed using a variety of accepted techniques.

3.2.1 Different types of grey fabric used

Cotton and Viscose grey fabric specification are mentioned in Table 3.3. The fabrics were supplied by different companies for the study.

Table 3.3 Cotton and Viscose rayon fabrics specification

Fabric	Type	Carded/ Combed	Pattern	Count	Width (in Inches)	GSM	GLM
Cotton	Woven	Combed	Plain	60s×60s	45.66	77	89.30
Cotton	Knitted	Carded	Rib	30s	55.9	150	212.90
VR	Woven	Combed	Plain	30s×30s	47.33	108	129.83

3.2.2 Different types of chemicals used

Table 3.4 Different types of products used for the process

Chemicals	Manufacturer
Caustic flakes	LR Grade (Sulabh lab)
Hydrogen peroxide (50%)	LR Grade (Sulabh lab)
Alpha amylase	VVM Chemicals Ltd.

3.2.3 Machines used for lab trial pretreatment process

For the application of treated fabric with EDA and PWA, various equipment were used. Name and make of the equipment are listed below.

Table 3.5 Instruments used and their manufacturer

Instrument Used	Application Area	Make of machine
Laundr-o Meter	Pretreatment of Desizing, Scouring and bleaching of Cotton woven, Knitted & Viscose woven fabrics	EEC pvt Ltd, Mumbai

Weighing Balance	Weighing purpose	Essae -Teraoka Ltd
pH Meter	Measuring pH of pretreatment solution	Analab Scintific Instrument pvt. Ltd
Oven	Drying of washed fabric	EEC pvt Ltd, Mumbai
Lab Instruments Like Beakers, Glass Rods, Spatulla, etc.	Different Application as per requirement	Borosil

3.2.4 Lab scale application method for pre-treatment of Cotton and Viscose rayon by modified process

In the present investigation pretreatment of Cotton and Viscose rayon has been experimented in Zydex Lab. Generally, pretreatment of both type of fabric is carried out by exhaust process or continuous process. The first goal was to take proper pretreatment of both fabrics in exhaust process. These pretreatment in exhaust processes on bulk scale are mostly done in soft flow machine for Cotton knitted and Viscose woven fabrics whereas Cotton woven done in Jigger/Jumbo/JT-10 machines. I have done simulating all three types of fabrics exhaust pretreatment processing in Laundr-o meter machine in Zydex lab. The current fabric pretreatment was also compared against the results obtained from the modified pretreatment by EDA and PWA in bulk scale.

3.2.4.1 Steps involved in the pre-treatment of Cotton woven in modified process

Wet processing steps involved in pretreatment of Cotton woven are Singeing, Desizing, Scouring, Bleaching and Mercerizing. In this work i have concentrate to developed product for Desizing, Scouring and Bleaching process only. That’s why focus on these three processes only.

Cotton pretreatment process consist of number of variables in terms of types of chemicals, concentration of chemicals, pH, temperature, time, water quality, etc. A number of experiments have been performed to narrow down parameters of Desizing and Combined scouring & bleaching.

The time of desizing, pH, temperature, auxiliaries' concentration, and other factors all affect uniform desizing. Numerous desizing experiments using EDA on cotton were carried out in an effort to optimize these parameters. Based on a strong Tegawa rating, the ideal desizing condition was chosen.

Variables taken for desizing process were

EDA (% owf)	:	0.3, 0.4, 0.5, 0.6
pH	:	4-5, 5-6, 6-7, 7-8
Temperature (˚C)	:	55, 65, 75, 85
Dwell time	:	30, 45, 60, 75
Water quality (ppm)	:	RO (20-30), Corporation (230-250), Borewell (590 & 850)

- **Optimization of bath pH**

Desizing was carried out using the exhaust approach while acetic acid was used to optimise the pH of the bath. Acetic acid was used to keep the acidic environment. It took place for 45 min in a laboratory shaking bath at a constant temperature of 75±2°C. The end outcome—a pH of desize liquor that was optimized—was chosen based on the Good Tegawa rating.

- **Optimization of bath temperature**

Experiments were carried out at various temperatures ranging from 55, 65, 75, and 85 ±2°C at optimized pH to optimize the temperature for desizing. The liquor ratio in the desize bath is 1:6, and it contains 0.5% (owf).

- **Optimization of dwell time**

Four baths with the same liquor ratio of 1:6 and optimized pH and desizing temperature were created in order to reduce desizing time. These baths were heated to an optimal temperature and maintained there for 30, 45, 60, and 75 minutes, respectively, to allow for desizing.

- **Optimum concentration of EDA**

To maximize the EDA's concentration during desizing. The only thing that changed in the optimized recipe's preparation of the dyebaths was the EDA concentration. The desizing was done at optimized pH, temperature, and time conditions as per the prior section, and the EDA concentration in the baths was 0.3, 0.4, 0.5, and 0.6% (owf), respectively.

- **Optimum quality of water for desizing**

In order to optimize time, temperature, pH and concentration for desizing, one of the major variables are water quality. Four baths were made using varied water qualities,

such as R.O. water, corporate water, and bore well water, each with a different pH value, time, temperature, and EDA concentration for desizing with the same liquor ratio of 1:6.

Bleaching and scouring together depends on a number of factors, including pH, temperature, auxiliaries' concentration, and duration. Several studies of combined scouring and bleaching with PWA on cotton were carried out in order to optimize these parameters. On the basis of good absorbency and a high whiteness index, the ideal scouring and bleaching conditions were chosen.

Variables taken for combine scouring & bleaching process were

PWA (% owf)	:	0.3, 0.4, 0.5, 0.6
NaOH flakes (% owf)	:	0.4, 0.6, 0.8, 1.0
Temperature (˚C)	:	80, 85, 90, 95
Dwell time	:	30, 45, 60, 75
Water quality (ppm)	:	RO (20-30), Corporation (230-250), Borewell (590 & 850)

- **Optimum concentration of Caustic**

To ensure that the bath's pH is optimal, combined scouring and bleaching was performed in presence of caustic by exhaust technique. It was carried out in laboratory by using all parameters on Optimize level and varies Caustic concentration. The final result was chosen as the optimized pH of liquor based on acceptable wetting and whiteness index.

- **Optimization of bath temperature**

Check optimum temp. of the bath by keeping all other variables at optimum level.

- **Optimization of dwell time**

Four baths were constructed with the same liquid ratio of 1:6 and optimised pH, temperature, and time for combined scouring and bleaching. These baths were heated to an optimal temperature and maintained there for 30, 45, 60, and 75 minutes, respectively.

- **Optimum concentration of PWA**

Baths were made according to the optimized recipe, with the concentration of PWA being the only change. PWA concentrations of 0.3, 0.4, 0.5 and 0.6 % (owf) were present in the baths, respectively, and the procedure was carried out under pH, temperature, and time conditions that were optimal.

- **Optimum quality of water for combined scouring and bleaching**

In order to optimize time, temperature, pH and concentration for combined scouring and bleaching, one of the major variables is water quality. Four baths were created using the same liquid ratio of 1:6 and optimized pH, duration, temperature, and PWA concentration. R.O. water, corporate water, and bore well water—all of varying quality—are used, respectively.

Above these variables were checked one by one in Desizing and combined Scouring & Bleaching process. After different permutation and combination, finalized a modified process recipe which has been taken bulk trial against current practice recipe in market.

Optimized Desizing for Cotton woven fabric (Recipe 1):

EDA (% OWF)	: 0.5
Acetic Acid (30%)	: To maintain bath pH
Bath pH	: 5-6
Temperature (˚C)	: 75 ± 2
Dwell time (mins)	: 45
Water quality (ppm)	: R.O.Water (20-30)
MLR	: 1:6

Optimized combined Scouring and Bleaching for Cotton woven fabric (Recipe 2):

NaOH (% OWF)	: 0.8
H_2O_2 (% OWF)	: 3
PWA (% OWF)	: 0.5
Temperature (ºC)	: 90 ± 2
Bath pH	: 13-14
Dwell Time (mins)	: 45
MLR	: 1:6

Procedure for pretreatment on Cotton woven fabric:

- Weighed the fabric for desizing process.
- Taken water in launder-o-meter container as per MLR.
- Added acetic acid to maintain the pH.
- Added EDA in above acidic water.

- Dip the fabric in container and started the procedure by automatic program feed (75˚C for 45 minutes).
- After completion, drained the liquor and squeezed the fabric.
- Added water in same container for scouring & bleaching process as per MLR.
- Added PWA, NaOH and H_2O_2 one by one in above water.
- Dip the fabric in above liquor, locked the container, put in launder-o-meter and start launder-o-meter with pre filled program (90˚C for 45 minutes).
- After completion of process, drained the liquor, rinsed the fabric in running water.
- Added fresh water for hot wash in container as per MLR.
- Start the procedure by automatic program feed (90˚C for 10 minutes)
- After completion of hot wash, drained the washing liquor.
- Added fresh water with Acetic acid for neutralization of fabric.
- Dry the fabric and tested this fabric as per standard.

3.2.4.2 Steps involved in the pre-treatment of Viscose rayon woven in modified process

Wet processing steps involved in pretreatment of Viscose woven are Singeing and Desizing. I have developed product for Desizing process.

Viscose pretreatment process consists of number of variables in terms of types of chemicals, concentration of chemicals, pH, temperature, time, water quality, etc. I have done range of experiments for narrow down parameters of Desizing process.

The degree of uniform pretreatment depends on a number of factors, including pH, temperature, auxiliaries' concentration, and desizing duration. With EDA on Viscose, numerous desizing trials were run in an effort to optimize these parameters. Based on a strong Tegawa rating, the ideal desizing condition was chosen.

Variables taken for desizing process were

EDA (% owf)	:	0.1, 0.2, 0.3, 0.4
pH	:	4-5, 5-6, 6-7, 7-8
Temperature (˚C)	:	55, 65, 75, 85
Dwell time (mins)	:	30, 45, 60, 75
Water quality (ppm)	:	RO (20-30), Corporation (230-250), Borewell (590 & 850)

- **Optimization of bath pH**

Desizing was carried out using the exhaust approach while acetic acid was used to optimize the pH of the bath. Acetic acid was used to keep the acidic environment. It took place for 45 minutes in a laboratory shaking bath at a constant temperature of 75°C. The end outcome - a pH of desize liquor that was optimized - was chosen based on the Good Tegawa rating.

- **Optimization of bath temperature**

Experiments were carried out at various temperatures ranging from 55, 65, 75, and 85±2°C at optimized pH to optimize the temperature for desizing. The liquor ratio in the desize bath is 1:6, and it comprises 0.3% (owf).

- **Optimization of dwell time**

Four baths with the same liquor ratio of 1:6 and optimized pH and desizing temperature were created in order to reduce desizing time. These baths were heated to an optimal temperature and maintained there for 30, 45, 60 and 75 minutes, respectively, to allow for desizing.

- **Optimum concentration of EDA**

To maximize the EDA's concentration during desizing. The only thing that changed in the optimized recipe's preparation of the dyebaths was the EDA concentration. EDA concentration in the baths was 0.1, 0.2, 0.3, and 0.4% (owf), respectively. The pH temperature and time parameters were optimized as described in the earlier section.

- **Optimum quality of water for desizing**

In order to optimize time, temperature, pH and concentration for desizing, one of the major variables are water quality. Four baths prepared with optimized pH, time, temperature and concentration of EDA for desizing maintaining liquor ratio 1:6 using different quality of water viz., R.O. water, corporation water and bore-well water.

Above these variables were checked one by one in Desizing process. After different permutation and combination, finalized a modified process recipe which has been taken bulk trial against current practice recipe in market.

Optimized Desizing for Viscose rayon woven fabric (Recipe 3):

EDA (% OWF)	:	0.3
Acetic Acid (30%)	:	To maintain bath pH
Bath pH	:	5-6
Temperature (˚C)	:	75 ± 2°C
Dwell time (mins)	:	45
Water quality (ppm)	:	R.O.Water (20-30)
MLR	:	1:6

Procedure for pretreatment on Viscose woven fabric:

- Weighed the fabric for desizing procedure.
- Taken water in launder-o-meter container as per MLR.
- Added acetic acid to maintain pH.
- Added EDA in above acidic water.
- Dip the fabric in container and start the procedure by automatic program feed (75°C for 45 minutes).
- After completion, drained the liquor and rinsed the fabric in running water.
- Added fresh water for hot wash in container as per MLR.
- Started the procedure by automatic program feed (85°C for 10 minutes).
- After completion, drained the washing liquor.
- Dry the fabric and tested this pretreated fabric as per standard.

3.2.4.3 Steps involved in the pre-treatment of Cotton knitted in modified process

Wet processing steps involved in pretreatment of Cotton knitted are Scouring & Bleaching. In the current study products have been developed for Scouring and Bleaching process.

Cotton pretreatment process consist of number of variables in terms of types of chemicals, concentration of chemicals, pH, temperature, time, water quality, etc. I have done range of experiments for narrow down parameters of combined scouring & bleaching.

The effectiveness of combined scouring and bleaching was dependent on a number of factors, including pH, temperature, auxiliaries' concentration, and time. On cotton knitted fabric, numerous tests of combined scouring and bleaching with PWA were

carried out in order to optimize these parameters. On the basis of good absorbency and a high whiteness index, the ideal scouring and bleaching conditions were chosen.

Variables taken for combine scouring & bleaching process were

PWA (% owf)	:	0.3, 0.4, 0.5, 0.6
NaOH flakes (% owf)	:	0.4, 0.6, 0.8, 1.0
Temperature (°C)	:	80, 85, 90, 95
Dwell time (mins)	:	30, 45, 60, 75
Water quality (ppm)	:	RO (20-30), Corporation (230-250), Borewell (590 & 850)

- **Optimum concentration of Caustic**

Optimization of pH of combined scouring and bleaching bath was performed in presence of caustic by exhaust technique. It was carried out in laboratory by using all parameters on Optimize level and varies Caustic concentration. The final result was chosen as the optimized pH of liquor based on satisfactory wetting and whiteness index.

- **Optimization of bath temperature**

The optimum temperature of the bath was chosen by varying the temperature and keeping all other variables at optimum level.

- **Optimization of dwell time**

Four baths were constructed with the same liquid ratio of 1:6 and optimized pH, temperature, and time for combined scouring and bleaching. These baths were heated to an optimal temperature and maintained there for 30, 45, 60, and 75 minutes, respectively.

- **Optimum concentration of PWA**

Baths were made according to the optimized recipe, with the concentration of PWA being the only change. PWA concentrations of 0.6, 0.8, 1.0, and 1.2% (owf) were present in the baths, respectively, and the procedure was carried out under pH, temperature, and time conditions that were optimal.

- **Optimum quality of water for combined scouring and bleaching**

In order to optimize time, temperature, pH and concentration for combined scouring and bleaching, the quality of the water is one of the key factors. Four baths were made using diverse water qualities, such as R.O. water, corporation water, and bore well

water, but with the same PWA concentration, pH, time, temperature, and liquor ratio of 1:6.

Above these variables were checked one by one in combined Scouring & Bleaching process. After different permutation and combination, finalized a modified process recipe which has been taken bulk trial against current practice recipe in market.

Optimized combined Scouring and Bleaching for cotton knitted fabric (Recipe 4):

NaOH (%OWF)	: 0.8
H_2O_2 (%OWF)	: 3
PWA (%OWF)	: 0.5
Temperature (°C)	: 90 ± 2
Bath pH	: 13-14
Dwell Time (mins)	: 45
MLR	: 1:6
Water quality (ppm)	: R.O. Water (20-30)

Procedure for pretreatment on cotton knitted fabric:

- Weighed the fabric for combined scouring and bleaching procedure.
- Taken water in launder-o-meter container as per MLR.
- Add PWA, NaOH and H_2O_2 one by one in above water.
- Dip the fabric in above liquor, lock the container, put in launder-o-meter and start launder-o-meter with pre filled program (90°C for 45 minutes)
- After completion of process, drained the liquor, rinsed the fabric in running water.
- Added fresh water for hot wash in container as per MLR.
- Started the procedure by automatic program feed (90°C for 10 minutes).
- After completion, drained the washing liquor.
- Added fresh water with Acetic acid for neutralization of fabric.
- Dried the fabric and tested this fabric as per standard.

3.3 Phase – III: Industrial scale application method for pre-treatment of cotton and viscose rayon by current process vs. modified process

A number of studies have been carried out as a part of a present investigation in the Zydex lab to decrease the amount of water and alkali used during the pretreatment

process, particularly when desizing, scouring, and bleaching cotton and viscose rayon. These laboratory investigations served as the foundation for large-scale industry testing (bulk trials) versus the standard operating procedure of that particular industry.

3.3.1 Different types of grey fabric used

As shown in table 3.3, many companies provided the grey fabrics. After an extensive amount of research on the fabric. Bulk trials were conducted using the same fabrics.

3.3.2 Different types of chemicals used

In the present investigations, industrial grade chemical products which are regularly used for textile pretreatment processes were used. In present study only generic name of auxiliaries is mentioned due to the non-discloser condition of brand name from the industrial supplies.

3.3.3 Machines used for bulk trial

Bulk scale trial for pretreatment of Cotton woven was done on Jigger machine where as other fabrics pretreatment were done in soft flow machine.

3.3.3.1 Soft flow machine

The soft flow machine is the best illustration of one of the techniques for circulating the cloth and liquor bath simultaneously. A soft flow machine is appropriate for treating a variety of knitted and woven fabric rope structures. This machine is very useful for the pretreatment & dyeing of lightweight woven fabrics.

Fabric loading and unloading system:

A continuous rope of cloth is used to circulate the material. To determine the continuous length of the available cloth to be dyed, the fabric's sections are stitched together. A woven cotton tape having good tensile strength is passed through the fabric passage during machine commissioning. Now, the upper end of the fabric is tied with the upper end of the cotton tape. The take-up roller or drive wheel carries the fabric inside the machine. The lower end of the cotton tape is pulled out slightly during loading till the upper end of the fabric comes out of the machine. The cotton tape is removed from the fabric now. The fabric loading gets continue till the lower end of the fabric. Now, the

upper and lower ends are stitched together to form an endless fabric. The fabric loading and unloading door is closed now.

When the fabric dyeing gets completed, the fabric is taken out of the machine through an unloading reel. The fabric is collected in the cloth trolley. At the end of fabric unloading, the cotton tape is inserted again in the fabric passage.

Dye autoclave or main dye vessel:

The dye autoclave is made of high-quality corrosion-resistant stainless steel. Basically, it is a high pressure closed vessel. The dye bath is filled in this autoclave. the fabric is loaded inside this autoclave. a leak-proof fabric loading and unloading door is mounted in front of the autoclave.

Nozzle or overflow tube and fabric transport system:

The rope of the fabric and dye liquor both move slowly in the soft flow fabric dyeing machine. The transportation of the fabric imparts a mild mechanical action. The dye liquor feeds to the nozzle at high pressure. The circulating fabric makes touch with the dye solution as it travels through the nozzle. Due to pressured liquid circulation, the dye liquor properly enters the fabric. The main dyeing vessel is a tubular vessel. The fabric coming out of the nozzle and narrow tube (delivery tube) falls into the main dye vessel. The falling fabric gets plaited continuously in the man dye vessel.

Figure 3.12 Ecotex Soft-flow dyeing machine **(Ecotex- next generation engineering)**

Liquor circulation pump and filter:

A high-pressure liquor circulation pump is used to feed the dye liquor into the nozzle. It takes the liquor from the main dye vessel and feeds it to the nozzle. A filter is also attached with a liquor pump that cleans the dye liquor continuously.

Heat exchanger:

A heat exchanger is mounted between the liquor circulation pump and the nozzle of the machine. The main function of the heat exchanger is to bring down the temperature when required. When the dyeing is completed, the cooling of the dye bath is performed with the help of this heat exchanger. **(Ecotex- next generation engineering)**

3.3.3.2 Jigger machine

In the history of the textile processing era, the jigger machine is a highly well-known and relatively straightforward machine. Although it places significant limitations on the processors, it is nevertheless commonly used despite its drawbacks.

The two main rollers of the jigger machines rotate on smooth bearings and are connected to a suitable drive mechanism that can be reversed as needed. One of the primary rollers is used to wind the fabric, and the other is used to feed it. The fabric passes through the dye liquor trough at the base of the machine as it moves from one roller to the next. At the bottom of the liquor trough, there are various arrangements of guide rollers, and during each passage, the cloth passes around these guide rollers.

Figure 3.13 Closed Jigger machine **(Mazharul, 2012)**

Typically, two equal amounts of the concentrated dye liquid are put immediately into the dyebath soon before starting the first and second ends. The fabric's passage through the dye bath stirs up the liquid. To speed up cloth rinsing, several horizontal spray pipes are installed across the entire width of the trough.

The liquor is heated by live steam that is pumped into the bottom of the trough through a perforated pipe that spans the width of the jig. Indirect heating is also provided via heat exchangers in some contemporary jigs.

The top of the jig should be covered to reduce heat loss to the atmosphere, maintain an even temperature throughout the fabric, and reduce air exposure to the alcohol and the fabric. When employing sulphur or vat dyes, it's crucial to limit exposure to the air because these colours can be oxidized by ambient oxygen.

To enable the whole length of the fabric to flow through the dye bath during the dyeing process, a few meters of leading fabric that is constructed similarly to the fabric being processed is attached to each end of the cloth batch. When the fabric has finished processing in the jig, excess water is removed during unloading by running it onto an A-frame using a nip or suction mechanism.

The drive, tension adjustment and control, fabric speed and metering, smooth and jerk-free stop and start, counters for number of turns, progressive and noiseless reversal, automatic temperature regulation and control, etc. of modern machines like automatic and jumbo jiggers are fully automated. **(Mazharul, 2012)**

3.3.4 Bulk scale application method for pre-treatment of Cotton and Viscose rayon by current process vs. modified process

3.3.4.1 Steps involved in the pre-treatment of Cotton woven in current vs modified process

Industrial trials were taken on 100% Cotton Woven fabric in Jigger machine in Surat unit. Basically, I have compared current process adopted by this industry with modified process with Polymer and enzyme mediated products for pretreatment of 100% cotton. Compared all aspects in terms of types of chemicals, heat energy, time, effluent load, etc.

Current procedure for pretreatment of Cotton woven fabric:

- Weighed the fabric rolls taken, stitched all takas & put in trolley.
- Taken water in Jigger machine tank. Heated it to 60 °C to remove impurities on fabric like physically attached soils, dust etc.

- Loaded the fabrics in stretch form through water by take up roller.
- On completion of loading of fabric by take up roller, it is known as first end.
- After completion of first end, take up roller became let off roller and then fabric was unloaded through same water tank by opposite roller called take up roller. This is known as second end process.
- After completion of second end, the machine automatically stopped. Liquor was drained.
- Fresh water was taken inside machine for desizing process. Added half quantity of below mentioned products one by one in water before each of next two ends started.
    - 0.2% (owf) Sequestering agent
    - 0.3% (owf) Acetic acid
    - 0.2% (owf) Defoamer
    - 0.8% (owf) Amylase Enzyme
- Raised temperature to 70 °C, and six ends were done.
- Drained liquor, added water for hot wash. Raised temperature to 80 °C, and two ends were done.
- Drained liquor, added water for scouring and bleaching process. Added half quantity of below mentioned products one by one in water before each of next two ends.
    - 0.2% (owf) Sequestering agent
    - 0.5% (owf) Wetting agent
    - 0.2% (owf) Defoamer
    - 2% (owf) NaOH
- Raised temperature of water to 60 °C, added half quantity of below mentioned products one by one in water before each of next two ends.
    - 3% (owf) H_2O_2
    - 1% (owf) Peroxide stabilizer
- Raised temperature to 95°C, done eight ends.
- Drained the liquor.
- Two hot washes at 95 °C each having four ends were done.
- Drained the liquor. Added water and neutralization with Acetic acid in final two ends were done.

- Checked Tegawa, absorbency and pH of fabric before unloading.

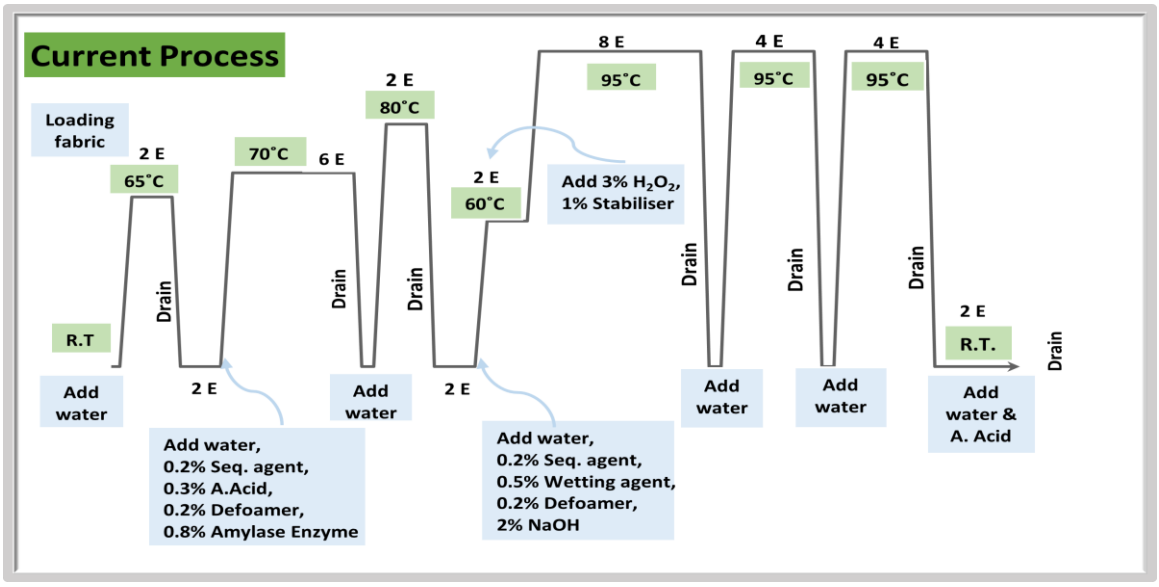

Figure 3.14 100% cotton woven fabric current process flow diagram in Jigger machine

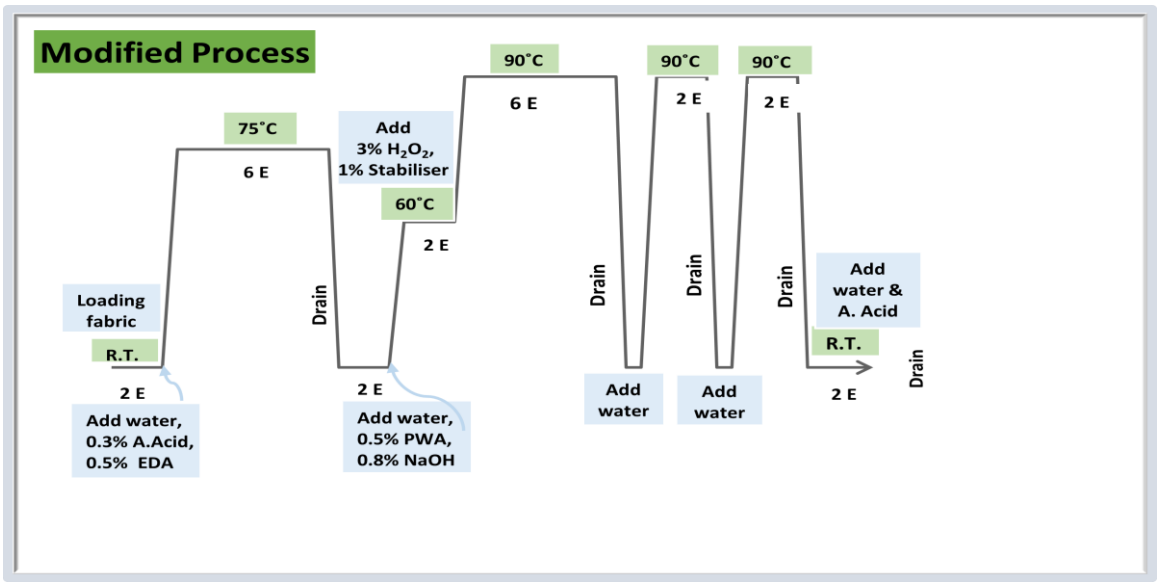

Figure 3.15 100% cotton woven fabric modified process flow diagram in Jigger machine

Modified procedure for pretreatment of Cotton woven fabric:

- Weighed the fabric rolls taken, stitched all takas & put in trolley.
- Taken water in Jigger machine tank for desizing process. Added half quantity of below mentioned products one by one in water before each of next two ends started.
    - 0.3% (owf) Acetic acid
    - 0.5% (owf) EDA

- Loaded the fabrics in stretch form through water by take up roller.
- On completion of loading of fabric by take up roller, it is known as first end.
- After completion of first end, take up roller became let off roller and then fabric was unloaded through same desize liquor tank by opposite roller called take up roller. This is known as second end process.
- After completion of second end, raised temperature to 75°C, done six ends.
- Drained the liquor.
- Added fresh water for scouring and bleaching process. Added half quantity of below mentioned products one by one in water before each of next two ends started.
    - 0.5% (owf) PWA
    - 0.8% (owf) NaOH
- Raised temperature up to 60°C, Added half quantity of below mentioned products one by one in scouring liquor before each of next two ends started.
    - 3% (owf) H_2O_2
    - 1% (owf) Peroxide stabilizer
- Raised temperature to 90°C, done six ends.
- Drained the liquor.
- Two hot washes at 90 °C each having two ends were done.
- Drained the liquor. Added water and neutralization with Acetic acid in final two ends were done.
- Checked Tegawa, absorbency and pH of fabric before unloading.

3.3.4.2 Steps involved in the pre-treatment of Viscose rayon woven in current vs modified process

In a Surat unit, commercial trials on 100% viscose-woven fabric were conducted. Basically, I have compared current process adopted by this industry with modified process with Polymer and Enzyme mediated product for pretreatment of 100% Viscose. Compared all aspects in terms of types of chemicals, heat energy, time, effluent load, etc.

Current procedure for pretreatment of Viscose woven fabric:

- Weighed the fabric rolls, stitched all takas & put in trolley.
- Taken water in soft flow as per MLR. & started loading the fabrics.

- Heated the water to 60 °C for removal of impurities on fabric like physically attached soils, dust, etc. Dwell time given was 10 minutes.
- Drained the liquor.
- Taken fresh water in to the machine for desizing process. Added below mentioned products one by one in water
    - 0.2% (owf) Sequestering agent
    - 0.4% (owf) Wetting agent
    - 0.2% (owf) Defoamer
    - 0.3% (owf) Acetic acid
    - 0.5% (owf) Amylase Enzyme
- Raised the temperature to 85°C and dwell time given was 60 minutes.
- Drained liquor, added water for two hot washes at 90 °C each of 10 minutes.
- Drained liquor, added water for cold wash at room temperature for 10 minutes.
- Drained the liquor.
- Checked Tegawa, absorbency and pH of fabric before unloading the same.

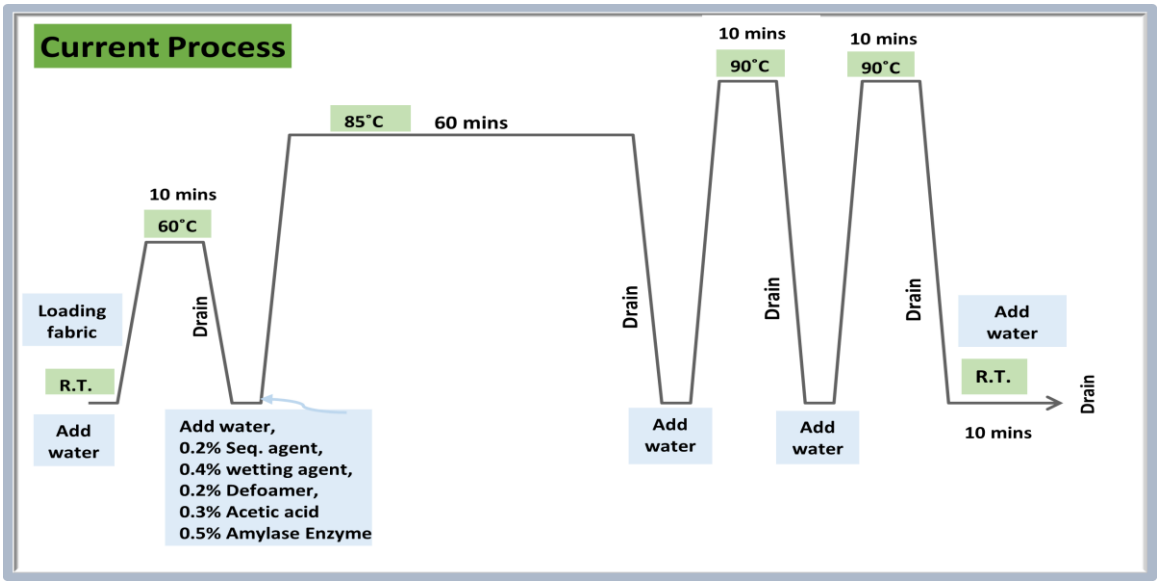

Figure 3.16 100% viscose woven fabric current process flow diagram in Soft flow machine

Modified procedure for pretreatment of Viscose woven fabric:

- Weighed the fabric rolls, stitched all takas & put in trolley.
- Taken water in soft flow as per MLR. & started loading the fabrics.
- Added below mentioned products one by one in water
    - 0.3% (owf) Acetic acid

- 0.3% (owf) EDA

- Raised the temperature to 75°C and dwell time given was 45 minutes.
- Drained liquor, added water for hot wash at 85°C for 10 minutes.
- Drained liquor, added water for cold wash at room temperature for 10 minutes.
- Drained the liquor.
- Checked Tegawa, absorbency and pH of fabric before unloading the same.

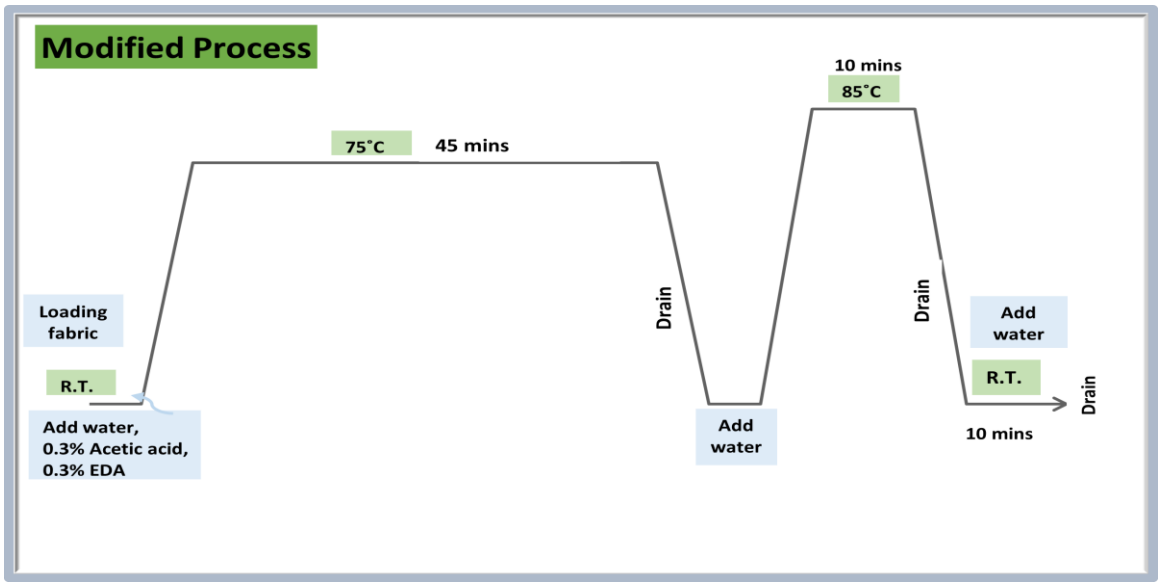

Figure 3.17 100% viscose woven fabric modified process flow diagram in Soft flow machine

3.3.4.3 Steps involved in the pre-treatment of Cotton knitted in current vs modified process

In the Surat unit, commercial experiments on 100% Cotton Knitted fabric were conducted. In essence, this study contrasted the pretreatment method now used by this industry with a modified method using a polymer-mediated product. Compared, all factors such as effluent load, time, heat energy, chemical kind, etc.

Current procedure for pretreatment of Cotton knitted fabric:

- Weighed the fabric rolls, stitched all takas & put in trolley.
- Taken water in soft flow as per MLR. & started loading the fabrics.
- Heated the water to 65°C for removal of impurities on fabric like physically attached soils, dust, etc. Dwell time given was 10 minutes.
- Drained the liquor.

- Taken fresh water in to the machine for combined scouring and bleaching process. Added below mentioned products one by one in water
    - 0.2% (owf) Sequestering agent
    - 1.0% (owf) Wetting agent
    - 0.2% (owf) Defoamer
    - 2.0% (owf) NaOH
- Raised the temperature to 60 °C and dwell time given was 10 minutes.
- Added below mentioned products one by one in water
    - 3.0% (owf) H_2O_2
    - 1.0% (owf) Peroxide stabilizer
- Raised the temperature to 95 °C and dwell time given was 60 minutes.
- Drained liquor, added water for two hot washes at 95 °C each of 10 minutes.
- Drained liquor, added water for cold wash at room temperature for 10 minutes.
- Done neutralization with Acetic acid in cold wash bath for 10 minutes.
- Checked Absorbency and pH of fabric before unloading the fabric.

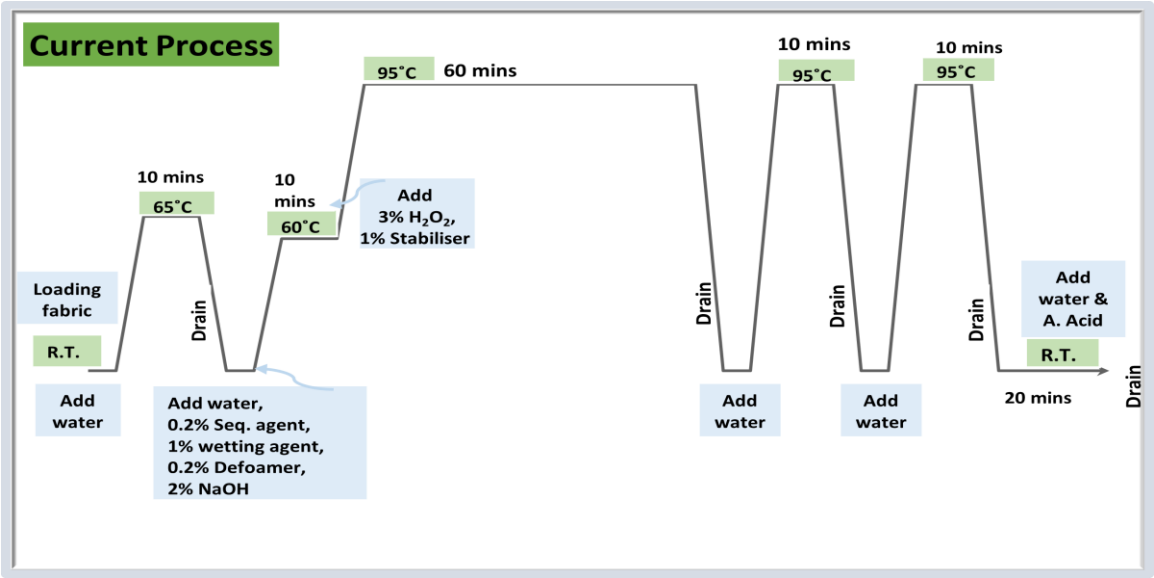

Figure 3.18 100% cotton knitted fabric current process flow diagram in Soft flow machine

Modified procedure for pretreatment of Cotton knitted fabric:

- Weighed the fabric rolls, stitched all takas & put in trolley.
- Taken water in soft flow machine for combined scouring and bleaching process as per MLR. & started loading the fabrics.

- Added below mentioned products one by one in water
    - 0.5% (owf) PWA
    - 0.8% (owf) NaOH
- Raised the temperature to 60 °C and dwell time given was 10 minutes.
- Added below mentioned products one by one in water
    - 3.0% (owf) H_2O_2
    - 1.0% (owf) Peroxide stabilizer
- Raised the temperature to 90 °C and dwell time given was 45 minutes.
- Drained liquor, added water for two hot washes at 90 °C each of 10 minutes.
- Drained liquor, added water for cold wash at room temperature for 10 minutes.
- Done neutralization with Acetic acid in cold wash bath for 10 minutes.
- Checked Absorbency and pH of fabric before unloading the fabric.

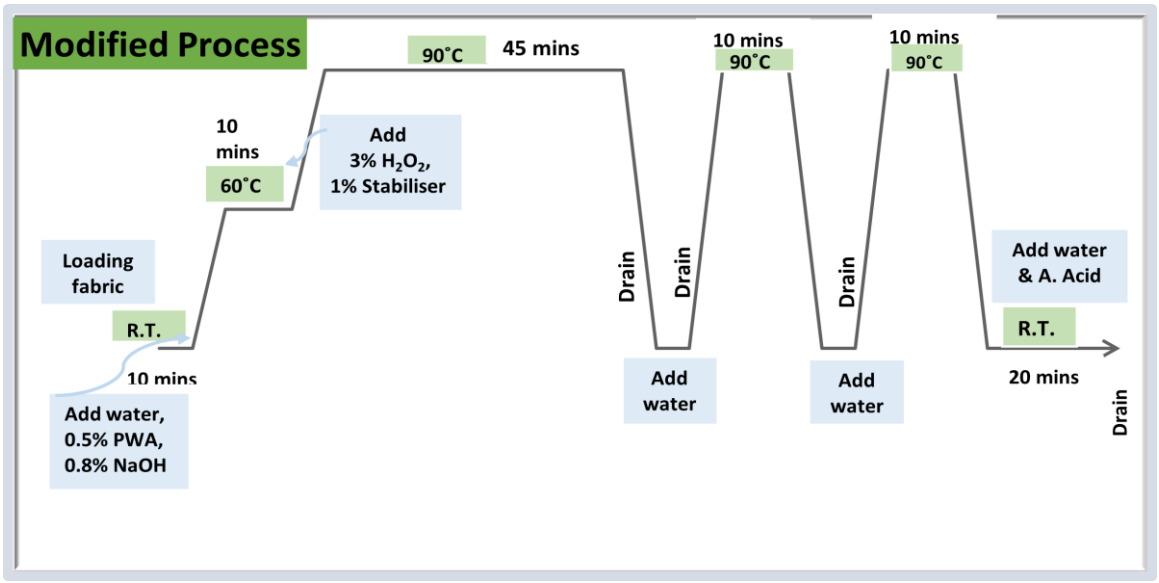

Figure 3.19 100% cotton knitted fabric modified process flow diagram in Soft flow

3.3.5 Testing method of pre-treated fabric

Higher customer satisfaction results from upholding quality standards. To do this, textile goods must pass a battery of meticulous inspections and tests throughout the wet processing step. In this work, standard testing methods for pretreated fabrics, commonly used by fabric pre-treatment industries were adopted.

3.3.5.1 Check weight loss of fabric

The fabric's starch is eliminated during desizing. The material will therefore lose weight once the operation is finished. This technique is also known as scaling

effectiveness. The desizing enzyme works more effectively the more weight is lost. The following formula can be used to calculate weight reduction in percentage terms:

% Weight Loss = [(Initial GSM – Final GSM) / (Initial GSM)] X 100

3.3.5.2 Tegawa rating

Preparation of reagent:

Prepare the reagent by mixing 10 gms of potassium iodide (KI) with 100 ml of water, then adding 0.6358 gm of iodine (100%), and stirring vigorously until the iodine is completely dissolved. Add 800 ml of ethanol after that. Then, the volume should be increased to 1000ml by adding water.

Method of testing:

Drop the aforementioned solution one or two times onto a piece of fabric. Gently rub it, and then evaluate the colour change using the Tegewa scale. The fabric should be cool and free of any remaining alkalinity before to testing.

Assessment:

No colour change indicates the absence of starch; pale blue to bluish violet = starch size is present, or a mixture of starch + synthetic size; Brown indicates the presence of modified starch or a starch/PVA size mixture. As shown below, it is a scale with colours ranging from violet to white.

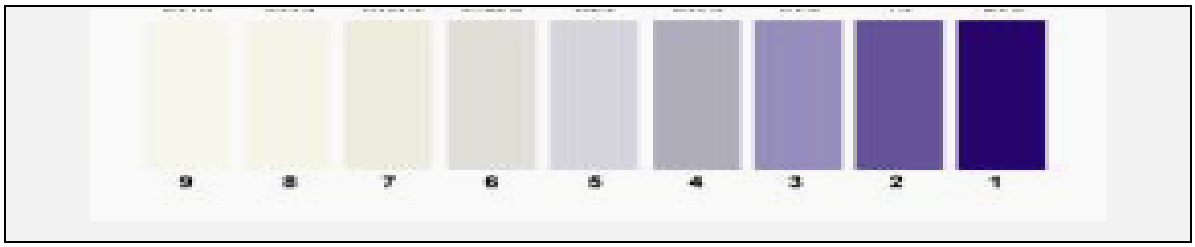

Figure 3.20 Tegawa scale

3.3.5.3 Measurement of absorbency

Drop test method:

The absorbency of pretreated fabric samples was measured using AATCC Test Method 79-2000. The test was run in a typical atmospheric setting. Two hours before the test, the samples were kept in a typical setting at 27 °C with a relative humidity of 65 ±2 %. The sample was initially put in an embroidery hoop to smooth out surface creases

without altering the fabric's structure. After that, the burette tip was placed inside the hoop, and the sample was exposed to one drop of distilled water.

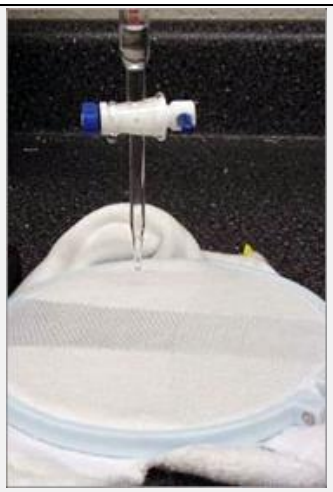

Figure 3.21 Drop test setup

A timer was also used to time how long it took for the liquid's surface to stop reflecting specularly. Each sample was subjected to five tests. This information is based on an average of five readings.

3.3.5.4 Sinking Time

The sinking method JIS L 1907: 2004 shall be as follows.

Apparatus:

For the apparatus, the followings shall be used.

1) Water bath, of such a size as it does not contact the specimens

2) Stopwatch, graduated by 0.5 sec.

Procedures:

Take three sheets of specimen of 10 mm x 10 mm size. Then, after floating the specimen so as to make the measured surface downward in the water bath containing water, measure the time until the specimen is wetted and starts to sink into water to the nearest 1 sec using a stopwatch. When one or two sheets among the three specimens do not sink, furthermore add three sheets of specimen and carry out the same operation. The test result shall be expressed by the mean value of three measurements of the time until the specimen is wetted and starts to sink into water by rounding off to an integer. When one or two sheets among three sheets of specimen do not sink, test another three sheets and express the mean value of the specimens which have sunk among the six sheets.

When the specimen does not sink after one hour or longer has elapsed, it shall be regarded as no sinking.

3.3.5.5 Whiteness and Yellowness Index

A spectrophotometer was used to test the whiteness and yellowness index of fabric samples (GretagMacbeth ColorEye XTS spectrophotometer; figure 3.23). The whiteness and yellowness indices were tested using the ASTM E313 method. All measurements were made with a 20° observer in big diameter and reflection mode. Check yellowness of the treated fabric by ASTM E313 in spectrophotometer with 5 scan, 4 fold

Each swatch was folded to four thickness and measurements were done on spectrophotometer with following specifications.

- Apparatus: UV spectrophotometer
- Model No.: Colour Eye XTS
- Manufacture: GretagMacbeth spectrophotometer CH 8105 Regensdorf, Switzerland
- Light of Source: Xenon arc lamp
- Range: 400-700 nm
- Software: colour iQc basic Version 9.4.40

The spectrophotometer was used to measure the bleached sample's whiteness index. The WI spectrophotometer was calibrated using white and black tiles prior to testing. There were 5 readings taken for each sample. And the outcome was revealed.

3.3.5.6 Core pH of fabric

pH of the Water-Extract from Wet Processed Textiles: AAATCC Test Method 81-2006

Specimens:

- Use a specimen of the test substance weighing 10 ± 0.1 g. The specimen should be cut into small pieces if it is difficult to wet out.

Procedure:

- For 10 minutes, slowly bring 250 ml of distilled water to a boil. Immerse the sample, and then boil for an additional 10 minutes with the beaker covered with a watch glass.

- Let the sealed beaker and its contents warm to room temperature. With tweezers, remove the specimen, letting any surplus liquid trickle back into the extract.
- Use a pH meter to measure the extract's pH while following the manufacturer's recommendations.

3.3.5.7 Dyeing of pretreated fabric and its colour strength

Reactive dyeing method for Cotton woven fabric in jigger machine:

- Loading the pretreated fabric and run for two ends in water
- Then added the pre dissolved dye in two parts and continued dyeing for four ends.
- Bottle green shade was taken for dyeing cotton woven fabric with following recipe:

  Corazol yellow RFT - 0.69

  Corazol Navy RFT - 1.10

  Coractive T. blue H2GP - 0.64
- Then salt was added in two parts. One part at room temperature for two ends.
- Raised the temperature to 50-55 ºC, second salt part added to run next two ends.
- Raised the temperature to 75-80 ºC, added alkali in two installments and continued for 4 - 6 ends.
- Drained the dye liquor and done cold wash for two ends.
- Souring was done with acetic acid at room temperature for two ends.
- Soaping done at 90ºC for 2-4 ends.
- Done two hot washes at 90ºC each of two ends.
- Cold wash and finally neutralized the fabric.
- Dried the fabric and checked colour strength in spectrophotometer.

Reactive dyeing method for Cotton knitted & viscose woven fabric in soft flow machine:

- Entered the pretreated fabric and run for 5 minutes.
- Then added the pre dissolved dye at room temperature and run for 10 minutes.
- Turquoise blue shade was taken for dyeing viscose woven fabric with following recipe:

  Corafix brilliant sky blue G - 0.26

  Corazol Yellow F3G - 0.15

  Coractive T. blue H2GP - 3.38

- Purple shade was taken for dyeing cotton knitted fabric with following recipe:

  Corazol yellow RFT - 0.24

  Corazol Red RFT - 0.96

  Corazol Blue RFT - 0.33

- Salt was added in two parts one part at room temperature and another part at 50 ºC run for another 10 mins.
- Gradually raised the temperature to 75-80 ºC
- Added soda ash and run for 40 -45 minutes.
- Drained the dye liquor and done cold wash for 5 minutes.
- souring was done with acetic acid at room temperature for 5 minutes.
- Soaping at 90ºC for 10 mins followed by two hot washes at 90ºC each of 10 minutes.
- Cold wash and finally neutralized the fabric.
- Dried the fabric and checked colour strength in spectrophotometer.

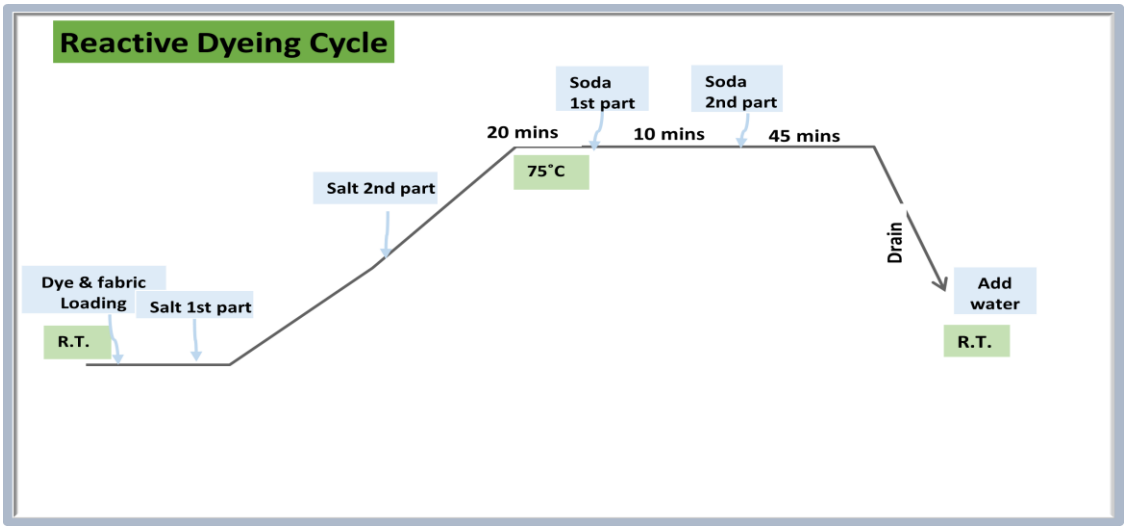

Figure 3.22 Reactive Dyeing Cycle

Colour strength:

Using a GretagMacbeth ColorEye XTS spectrophotometer (Manufacturer: X-rite Pantone, USA), the colour strength values of all six fabrics, including cotton woven, cotton knitted, and viscose woven, with the original and modified pretreatment processes, were measured.

A spectrophotometer was used to measure the *DE and strength* of the coloured sample. The spectrophotometer was calibrated with white and black tiles prior measurements. For each sample, 5 readings were collected, and the results were reported.

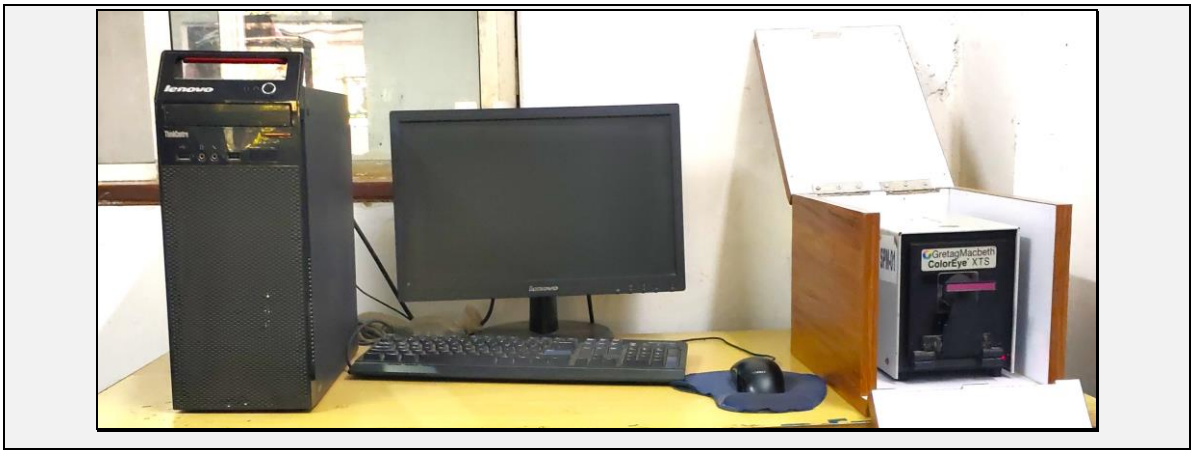

Figure 3.23 Colour Eye Spectrophotometer

3.3.5.8 Tensile Strength Measurement

Use a tensile strength tester to determine the breaking load of fabric samples (LRY Model, Lloyd, U.K.). The testing was done at the Textile Engineering Department of the M.S. University of Baroda. Prior to physical testing, the samples were dried and condition at a temperature of 27±2 °C and a relative humidity of 65±2%.

Determination of tensile properties

Tensile strength is a measure of a textile's durability expressed in the number of pounds of force per square inch needed to break a yarn or fabric. The force exerted on a sample at the moment of failure, which is often the greatest force, is referred to as the tensile strength. The force or load needed to rupture a material is referred to as its tensile strength. The textile industry uses a variety of different units to convey strength around the globe. Basically, pounds per square inch is the measurement of fibres that is most frequently used in English-speaking regions.

The ability of a material to withstand the strain caused by tensile loading is known as tensile strength. A structural element experiences a pulling force when it is subjected to tensile loading. At the point of stress, the cross-sectional area A is equal to the magnitude of the force acting on the elements, 7, expressed in pounds, divided by the unit stress. S = 7/A thereby. Materials have a wide range of intrinsic strengths. The designer must be careful when choosing structural materials to ensure that the tensile stress the material will experience does not surpass its ultimate tensile strength.

Tensile strength is the capacity to withstand breaking due to tension or pulling. The force per unit of cross-sectional area, or Kg/m2, is a common way to express it. Tenacity, the most popular term for fibre and yarn strength, is the force per unit of linear

density; however, tensile strength and tenacity are not the same when the yam number is given as an inter-unit length, such as gramme per denier. Wet strength is frequently a crucial quality, especially in vegetable fibres, which have more wet strength than their dry breaks, and in rayon and protein fibres, which typically have lower wet-to-dry tenacities.

It should be noticed that each of these physical characteristics somewhat influences property number, which has to do with the spinnability of a fibrous material. All prospective fibres need not have all of these characteristics, but specific spinning procedures can make up for some of the factories that a certain fibre may lack. In addition to specification and use, there are additional physical qualities that affect the production of yarn and the uses of the fabric or clothing.

At a traversal speed of 300 mm/min, 2 inch × 12 inch cloth samples were examined to determine the breaking load, breaking elongation, stress, and strain. B.S. 2576:1959 was followed when performing the test. (B.S. Handbook No. 11, P. 249)

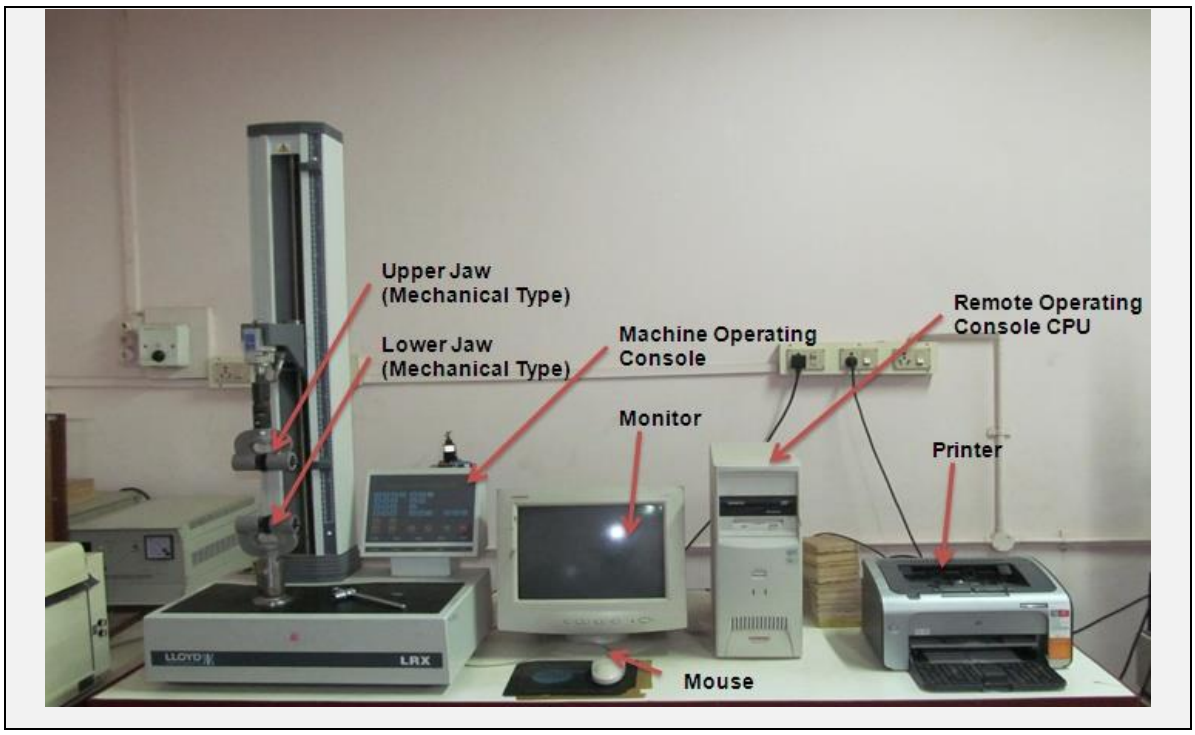

Figure 3.24 Lloyd tensile strength testers

3.3.5.9 Tear Strength Measurement

The strength of the yarn and the makeup of the fabric are the main determinants of tensile and tear strengths. Low individual fibre tenacities can be made up for by strong yarn or fabric structures, hence the fibre tenacity is comparatively less significant.

Tensile strength is less obviously associated with the usefulness of woven materials than tear strength. When testing tensile strength, the stress applied to each yarn

is the same. To the greatest extent possible, only a few yarns are stressed when it comes to tear strength. The highest tear strength will be found in fabrics manufactured by weaving together groups of threads since more yarns will join together to share the load. Tenacity, which is expressed in units of force per unit of sample cross sectional area, is the sample's breaking strength. It is the yarn's breaking strength, measured in grammes per denier.

Greatest longitudinal stress that a material can take without cracking. opposition to rupture. It is sometimes expressed as the number of kilogram's or pounds per square centimeter needed to produce rupture relative to a cross sectional area of one unit. The American Society for Testing and Materials refers to the durability of textile fibers as their tenacity. The force required to rupture or break the fibre is used to calculate tenacity. Fiber tenacity is assessed in either games per tex or games per denier.

According to ASTM D1424-09, the Elmendrof tearing tester was used to determine the fabric's tear strength. This instrument's pendulum has a sector shape, as seen in Figure 3.25. The sample is held in place by two clamps, one fixed and the other mobile. The movable clamp separates from the fixed clamp when the pendulum is released and the fabric tears across the breadth. The tearing force is displayed as a pointer in gms.

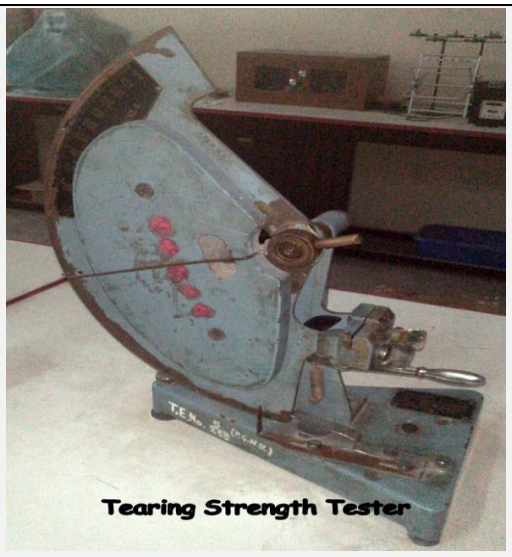

Figure 3.25 Elmendrof tear strength tester

3.3.5.10 Feel of pre-treated fabric by subjective evaluation

Feel of fabric is checked manually. It should be comparable to standard with a variation of ± 10%.

3.3.6 Testing method for effluent solution, discharged from pre-treatment department

The Textile processing sector is water intensive and water plays a major role in all areas such as heating, cooling and wet processing. The process also produces considerable amount of waste water, which is polluted by the use of dyes and auxiliaries. Environmental concerns are not only threatening the textile industry, but the entire chemical industry. The process being heterogeneous phase reactions, complete removal of dyes and chemicals is highly impossible. The lack of good water quality stringent demands and increasing water cost make good Waste Water Management or Effluent Management is must.

The term "environmental pollution" refers to all the ways that individuals pollute their surroundings. Unwanted changes to the environment's physical, chemical, or biological properties have negative effects. One of the most important issues facing humanity is environmental pollution. The government must create some laws and regulations that require businesses and individuals to stop or scale back on certain polluting activities. Environmental regulators are paying more attention to textile wet processing facilities because of the complicated wastewaters and air pollutants they produce. This new legislative initiative comes at a time when textile companies are already under pressure to cut costs in order to keep up with the fiercer competition. Textile industries have the chance to limit pollutant discharges and make financial savings through pollution avoidance.

For a textile processing operation, it's critical to precisely identify each source of waste. Inventory control, understanding of potential contaminants in purchased materials, and process analysis can all help with this. This is taken into consideration as we study and report on washing effluent parameters including pH, TDS, BOD, and COD for polymer & enzyme treated textiles.

3.3.6.1 Determination of pretreated effluent TDS

Total dissolved solids (TDS) are the phrase used to describe the sum of all inorganic and organic substances that are suspended in a liquid as molecules, ions, or microgranules (colloidal sol). The TDS of the wastewater was determined using a portable conductivity TDS meter.

According to the operational definition, the solids must be so small that they could pass unharmed through a sieve with a mesh size of two micrometres. Total dissolved

solids are typically only discussed in connection to freshwater systems since salinity includes some of the ions that are used to define TDS. TDS is used as a measure of the aesthetic qualities of drinking water and as an all-encompassing indicator of the presence of a wide range of chemical contaminants in streams, rivers, and lakes even though it is not generally regarded as a primary pollutant (i.e., it is not deemed to be associated with health effects).

Figure 3.26 TDS meter

3.3.6.2 Determination of pretreated effluent BOD

BOD is a gauge of how effectively water pollutes. In order for inorganic and organic particles to be oxidised in the effluent, oxygen is necessary. BOD, a measure of the oxygen demand by organic matter, is the quantity of oxygen required to complete the biological breakdown of dissolved solids in aerobic conditions at standard temperature. Despite being commonly employed as a gauge of water's organic quality, it is not a precise quantitative test. The most popular unit of measurement for organic water pollution is milligrammes of oxygen consumed per litre of sample. **(Ananthashankar, 2013; Gujarat Pollution Control Board; Effluent Standard)**

BOD is measured using the dilution method. Five ml of sample, one ml of waste effluent, one ml of each of the four standard buffers—magnesium sulphate buffer, calcium chloride buffer, phosphate buffer, and ferric chloride buffer—as well as distilled water to dilute the sample—were all added to a 500 ml BOD bottle. Iodometric titration

was used to rapidly identify the dissolved oxygen (blank, A). A second sample was made as described above, incubated at 27 °C for three days, and the dissolved oxygen content was then determined (sample value, B).

For three days, the sample is held at 27 °C and in the dark to halt photosynthesis (and the subsequent addition of oxygen), after which the dissolved oxygen is once more measured. The BOD distinguishes the final DO from the original DO. To obtain the corrected value, the apparent BOD for the control is deducted from the control result. The BOD is the amount of dissolved oxygen that has been lost in the sample after the degree of dilution has been adjusted for. Then, using Eqn 1, the BOD (in mg) was calculated.

$$BOD = (A - B) \text{ X dilution factor} \quad (1)$$

3.3.6.3 Determination of pretreated effluent COD

It serves as a gauge for the amount of oxygen needed by a sample that is vulnerable to oxidation by potent chemical oxidants. Due to its greater oxidizing power, adaptability to a wide range of samples, and simplicity of use, the dichromate reflux method is chosen over processes utilizing other oxidants (such as potassium permanganate). The majority of organic compounds oxidize between 95 and 100% of their theoretical value.

When using a dichromate in an acid solution to oxidize unstable components in a sample, COD is the amount of oxygen needed. A 250 ml conical flask was filled with 50 ml of the sample, 1 g of mercury (II) sulphate, 180 ml of silver sulphate solution, and sulphuric acid. The resultant combination was then added to 10 ml of 0.00833 M standard potassium dichromate solution, and it was heated for 15 minutes. After cooling, 50 ml of water were used to rinse the condenser's inner surface. Diphenylamine or ferroin indicators (1 ml each) were added, and 0.025 M ammonium iron(II) sulphate solution was used to titrate the results. At the terminus, diphenylamine changes colour from blue to green, whereas ferroin changes colour from blue-green to red-brown. The back-titration is repeated for the blank (B ml), which is referred to as titration A ml. The amount of potassium dichromate consumed during the oxidation is what causes the discrepancy between the two values. The COD (in mg) was then calculated using Eqn 2.

$$COD = (A - B) \text{ X } 0.2 \text{ X } 20 \text{ mg } l^{-1} \quad (2)$$

Procedure to check COD:

In a 500 ml refluxing flask, add a 50 ml sample or an aliquot that has been diluted to 50 ml. In order to prepare the blank, 50ml of distilled water is used. Use of a 50ml volumetric pipette is recommended because this measurement must be exact. Include 5–7 glass boiling beads. 5 cc of concentrated sulphuric acid/silver sulphate solution, 1 g of mercuric sulphate ($HgSO_4$), and mix until the $HgSO_4$ is dissolved. The mercuric sulphate's job is to complicate or bind chlorides. In cases where the chloride concentration is low, one gram may not be necessary. (Caution: To prevent overheating, add acid into the flask gently while mixing. Because of the heat produced, gloves may be required.) Add 25ml of 0.25 N potassium dichromate ($K_2Cr_2O_7$) precisely, and then stir. 70ml more of the concentrated sulphuric acid-silver sulphate solution should be added while mixing. Attach the flask to the reflux condenser after thoroughly mixing, then heat and reflux for two hours. The length of the refluxing process can be shortened depending on how quickly organic molecules oxidize. By refluxing for intervals ranging from 15 minutes to 2 hours and comparing the results, this duration can be calculated. With each set of samples, a reagent blank containing 50ml of distilled water treated with the same reagent as the sample should be refluxed. After the refluxing period, cool the device to room temperature. Condenser and flask interiors should be cleaned twice with around 25ml pieces of distilled water. Take the flask out of the condenser and add distilled water to dilute it to a final amount of about 350 ml. Add a magnetic stirring rod and 4 to 5 drops of ferroin indicator. Titrate the flask quickly with 0.1 N ferrous ammonium sulphate to the first red-brown endpoint while it is mounted on a magnetic stirrer. Titrate with caution. The endpoint can be quickly achieved and is quite acute. **(Ananthashankar, 2013; Gujarat Pollution Control Board; Effluent Standard)**

3.3.6.4 Determination of pretreated effluent pH

In chemistry, pH serves as a gauge for a solution's acidity or basicity. At 25 °C (77 °F), pure water is said to have a pH level close to 7.0, making it neutral. Acidic solutions are those with a pH under 7, and basic or alkaline solutions are those with a pH over 7. **(Ananthashankar, 2013; Gujarat Pollution Control Board; Effluent Standard)**

An accurate measure of pH obtained by using a pH meter which is shown in figure 3.6