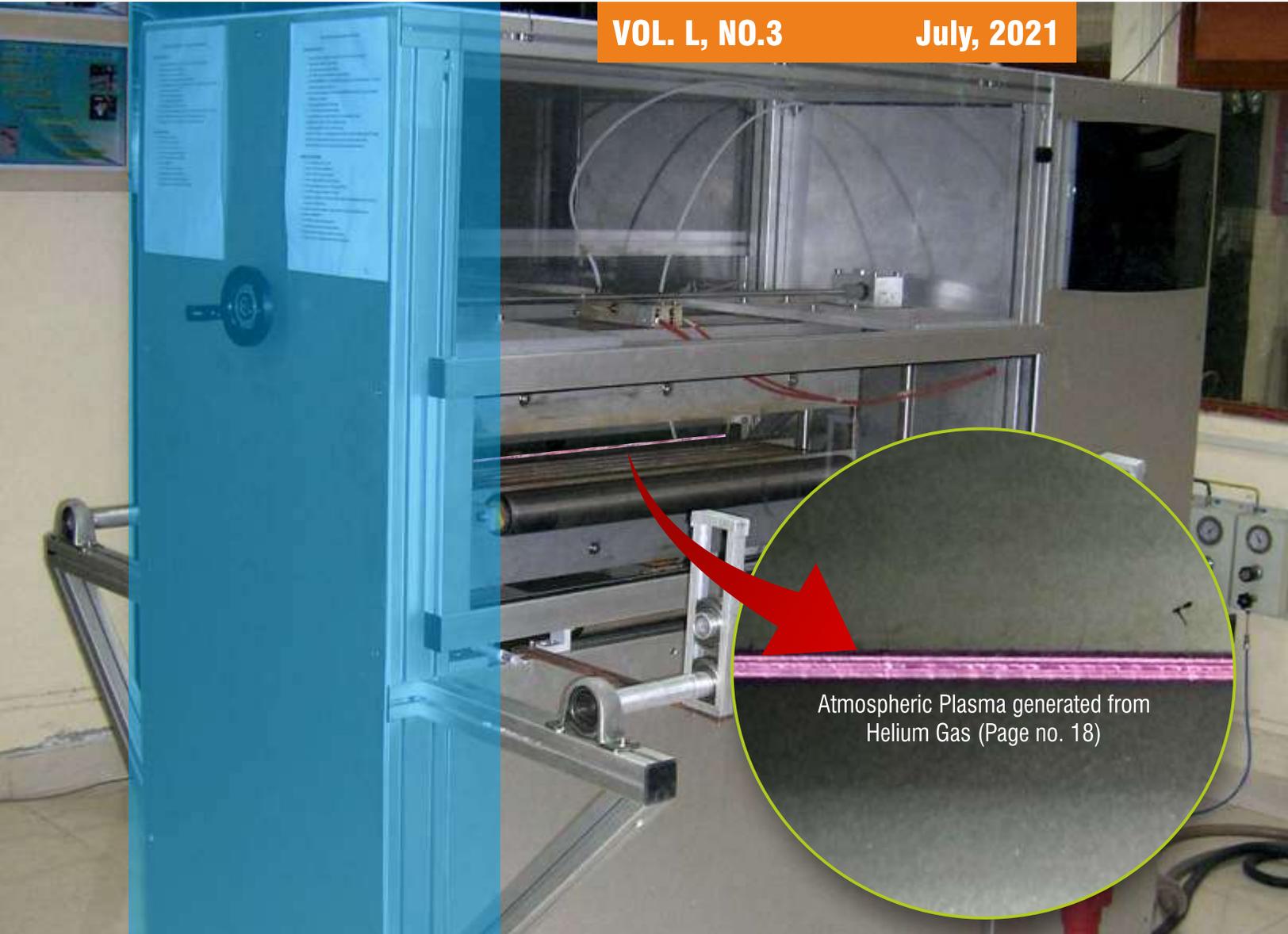


btra scan

VOL. L, NO.3

July, 2021



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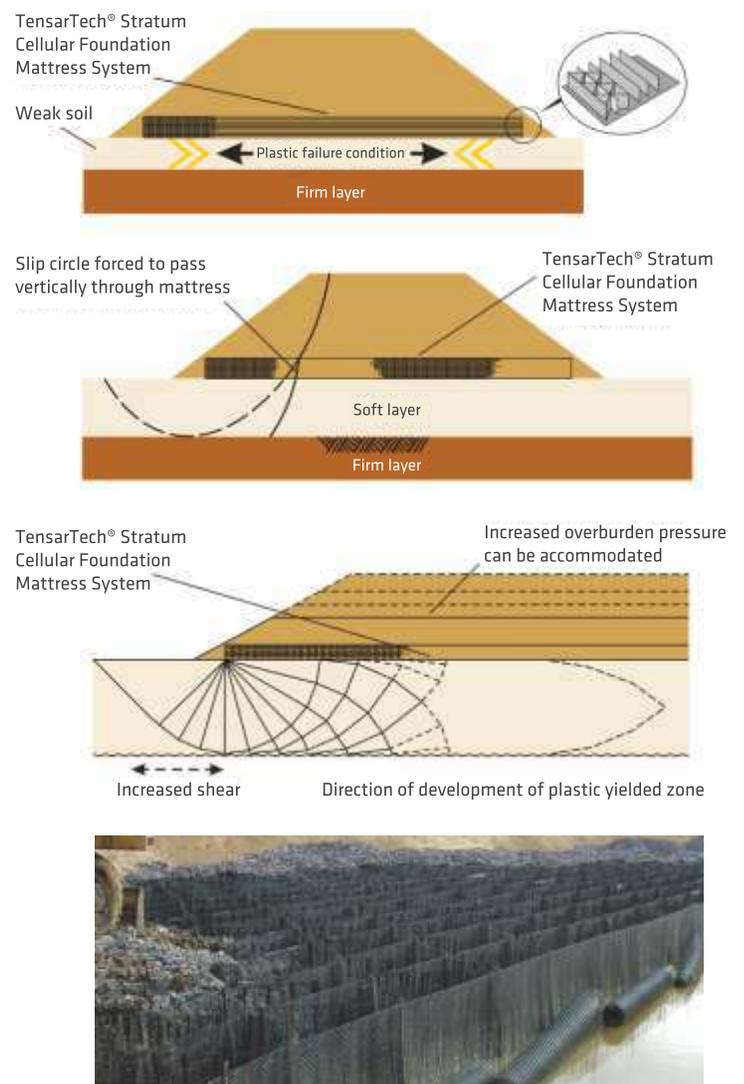
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EDITOR'S DESK

Dear Readers,

Greetings!!

Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. In continuation to this effort, I am delighted to present to our readers the 52th Edition of BTRA SCAN. After a year and half, still we are struggling with the pandemic and facing many challenges. However, we have to focus on our progress accepting the challenges and difficulties.

This issue has papers from the different domains such as natural dye, chemical testing and effect of plasma treatment to improve adhesion. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

The future is undoubtedly positive for the industries. I feel we will have a great time ahead after recovery." Hope, we all are following the safety practices to defeat the pandemic completely from our life soon.

Our sincere thanks to all the reader and contributors for their support and interest.

Dr. T V Sreekumar
Director, BTRA

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Editor : Dr. T. V. Sreekumar, BTRA
Associate Editor : Dr. Prasanta Kumar Panda,
BTRA

ISSN 0972 - 8341

BTRA Scan is abstracted by -

- Elsevier Bibliographic Database, UK,
- EBSCO Research Database, USA
- Chemical Abstract Services, USA

BTRA Scan is published quarterly by the Bombay Textile Research Association, Lal Bahadur Shastri Marg, Ghatkopar (W), MUMBAI — 400 086, INDIA; Tel. : 022-62023636; Email : info@btraIndia.com Website: www.btraIndia.com

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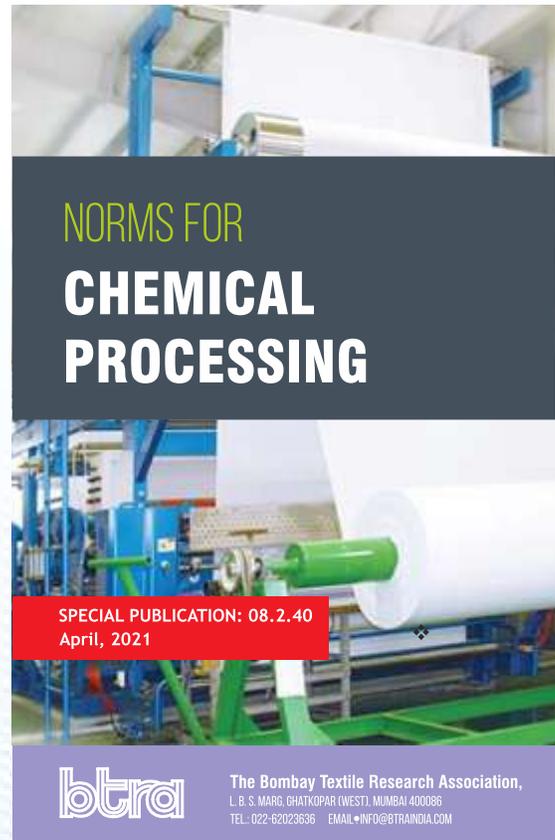
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Zero Liquid Discharge ETP – A Case Study (Part II)

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Abstract

The various treatment steps required to get the complete textile ZLD ETP were already discussed. Primary, secondary and tertiary treatments followed by advanced filtration are the important steps of ETP. Understanding the principles of each process, effluent retention time in each step, and the necessity of proper sizing of equipment are the main key factors and performance assuring things in ETP. The filtration system and TDS mass balance along with permeate water quality is discussed with facts and figures.

Keywords

Textile, ZLD, ETP, Effluent retention time, primary treatment, secondary treatment, filtration system, TDS mass balance, activated carbon filter, reverse osmosis

1.0 Introduction

In part I of this article series, we have understood the characteristics of the influent and basic effluent treatment scheme. Here, as we are discussing a case study of 600KLD (600000 liters of the effluent/day) effluent from a yarn dyeing plant. The described treatment scheme is as per the case study and plant provided by us. A complete treatment scheme to achieve Zero liquid discharge was shown and the theoretical aspects, the principle of each treatment, and the information on processes were shortly described in part I already. Now, in part II of this article series, We will be discussing the details about each component of ZLD ETP.

2.0 Components of ZLD plant

ZLD ETP plant consists of four major parts i.e. Primary treatment, Secondary treatment, Tertiary treatment, and advanced filtration with salt recovery to get ZLD. Each part is explained below.

2.1 Primary treatment process and it's component

Mainly primary treatment consists of the following component

- Bar screen
- Oil and grease trapper
- Collection tank
- Equalization tank or homogenization tank
- Anaerobic digester (may be recommended for COD > 2500 ppm

- Flash mixer with mixing channel and dosing system
- Primary clarifier

The Working and purpose of these components are explained below;

- ❖ Here, we are treating the effluent coming out from the yarn dyeing process. The yarn dyeing process takes place at higher temperatures around 90-95°C.
- ❖ Effluent from process house contains high temperature and variable in pH Value.
- ❖ Two different raw effluent storage tanks are provided called collection and Equalization tanks.
- ❖ Before the collection tank, one screening system is provided called Bar Screen, Oil & Grease Trap.
- ❖ Bar screen - to screen flubs, dust, other heavy metals from the process house.
- ❖ Oil & Grease Trap – generally oil & Grease having lower density nature, it will float over effluent. We can easily screen oil & Grease with Over Flow tee pipes.
- ❖ The necessity of oil & Grease removal – without removal of Oil & Grease, it forms a layer over effluent and prevent contact between atmospheric air and water. It will lead to reducing the effluent treatment process efficiency.
- ❖ Selection of Collection & Equalization tank capacity:
 - Generally, the exhaust yarn dyeing process takes a maximum 6-8 hours per batch.

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- Thus, on of average 3 batches per day are dyed. Here, in this case, due to high temperature, the collection and equalization tanks are provided with the capacity of 12 Hours retention time.
- The coarse bubble diffusion mechanism is provided in the collection tank for temperature reduction as well as effluent homogenization.
- ❖ Raw Effluent transfer pump and Selection
 - Here, in this case, the effluent transfer rate is calculated based on 20 Hours of operation and head calculated from vertical sump depth of collection tank and anaerobic tank height from ground level.
 - Equalization blower and diffuser selection are based on tank volume, Oxygen transfer and type of bubbling. We have provided a snap cap diffuser for the higher oxygen discharge rate.
- ❖ Acid Dosing System
 - Before the anaerobic inlet, we need to correct the pH to the range of 7, because bacteria development achieves only in a neutral base.
 - Hydro chloric acid dosing is given for pH correction.
- ❖ Anaerobic Digester tank
 - This treatment is in the absence of oxygen and is used where COD values are very high i.e. >2500
 - we have given a retention time of 48 hours for the anaerobic process to reduce COD, BOD and Colour removal up to the limit.
 - The feeding of cow dung, enzymes will increase the population of bacteria in an anaerobic tank. It will lead to getting better efficiency in COD, BOD reduction.
- ❖ Chemical treatment system
 - We have provided one mixing channel system along with the flash mixer setup for better agitation.
 - Primary treating chemicals are lime, ferrous for colour removal and precipitation, poly for the sludge settling process.
 - alternately we can use CRP, Alum for colour removal and precipitation, poly for sludge settling. Consumption of CRP and Alum will reduce the sludge generation in the ETP system.
 - Low HP installed Chemical mixing agitators with a speed ratio of 25:1 provided based on chemical consumptions for chemical mixing.
- ❖ Primary Clarifier
 - Flocculators having a speed ratio of 40:1 are provided for better agitation in the flash mixer.
 - Flash mixer is having a capacity of 20 Minutes retention time of average daily flow.

- A primary clarifier system is provided for the settling of suspended solids and turbidity, it is designed based on flow velocity and surface area.
- Primary clarifier is equipped with MS Bridge, gear box with a speed ratio of 70:1, worm reduction drive, center shaft, A frame, Scrubber arm, chequer plates and accessories.
- Sludge from the primary clarifier will discharge out to sludge thickener for the sludge handling process.
- Sludge handling will do with the help of a decanter, Screw press, or filter press to convert sludge into dry sludge.

To achieve the better efficiency of pollution removal from the effluent, the hydraulic retention time (HRT) of every process is important and they are as mentioned in below Table 1.

Table 1: Component wise HRT (hours), parameter control and purpose

Sr. No.	Component	HRT	Purpose of treatment	Critical parameters
1	Bar screen chamber	Online	To screen flubs, dust, other heavy metals	
2	Oil grease trapper	Online	Floating oil and grease screening	
3	Collection tank	12 Hrs	To collect the effluent from the process	Ensure no bigger suspended solids and fibrous material in it
4	Equalization tank	12 Hrs	Proper mixing and homogenization of the effluent	Temperature < 38°C with the help of aeration, pH 7-8 by HCL dosing
5	Anaerobic digester	48 Hrs	To reduce colour, COD and BOD	Temperature - < 38 pH - 7.5 to 8
6	Flash mixer	20 minutes	To coagulate and flocculate the suspended solids/pollutants	Based on jar test, optimized Dosing of PAC, decolourant and polyelectrolyte
7	Primary clarifier	6 Hrs	Settling of suspended solids as sludge	Flocculator speed ratio

2.2 Secondary treatment process and its component

This treatment is mainly biological and carried out in presence of oxygen.

2.1.1 Biological Aeration tank

- Biological aeration is also called an activated sludge process and is the Heart of the ETP Process, where the COD and BOD reduction takes place up to 90%.
- Here the aeration tank is designed with a retention time of 36 Hours.
- Aeration blowers with fine bubble diffuser provided for better oxygen transfer efficiency.
- Generally, the Standard oxygen transfer efficiency rate is only 23% from atmosphere air and only 18% of them were utilized for MLSS development. The remaining 88% is considered as a loss.
- Identifying of good aeration process is based on MLSS level and DO level.
 - Taking a jar of 1000 ml effluent from the aeration tank contains 300-400 mg of MLSS will be considered as good aeration efficiency.
 - Maintaining DO in between 1-2 ppm.
 - Maintaining the temperature between 25-38 Deg Celsius.

2.1.2 Secondary Clarifier

- The secondary clarifier is mainly designed to re-circulate aged bacteria to the aeration tank.
- The design of the mechanism is similar to the primary clarifier mechanism.
- The flow rate of recirculation bacteria is kept at half of the average daily flow rate.

2.1.3 Addons

- ❖ Change Over for Aeration blower
 - It is used to change each blower for an operation of 10 Hours per day cycle.
 - Auto ON/OFF Control.
- ❖ Online DO Meter
 - Dissolved Oxygen meter. (DO) : It is used to measure the level of DO in the aeration tank to increase or decrease the oxygen rate with the help of VFD Control Drives.
 - Case-1: DO level < 1: Need to Increase oxygen transfer efficiency by increasing Hz in VFD control.
 - Case-2: DO level >2 : Need to reduce oxygen transfer efficiency by decreasing Hz in VFD Control.
 - VFD controlling will be done automatically with the DO meter analyzer.

- ❖ pH, TDS meter for regular parameter analysis.
- ❖ Electromagnetic flowmeter : To measure average flow per hour.

The hydraulic retention time (HRT) and critical parameter to be controlled in the biological tank (secondary treatment) are important and they are as mentioned in the below table 2

Table 2:- HRT and critical parameter to be controlled in the biological tank

Sr. No.	Component of treatment	HRT in hours	Purpose of treatment	Critical parameters to be controlled
1	Aeration tank	36 Hours	To reduce Cod and BOD with the help of bacteria	DO level 1-2 pH- 6.5 – 7.5 Temperature < 35°C MLSS - 3000 to 4000 ppm
2	Secondary clarifiers	6 Hours	To clarify the water	
3	Holding tank	24 hours	To hold treated water	-

2.3 Tertiary treatment

- Tertiary treatment is like river water treatment; here we can control individual parameters like hardness, colour removal, Silica, Suspended solids and turbidity control.
- Chemical mixing is based on the type of treatment to be obtained.
- Caustic and Soda ash will reduce hardness.
- Alternately we can use Lime and Soda Ash for hardness control. It is named lime soda softening.
- Lime/CRP, Ferrous sulphate/ALUM/PAC, and Poly for Colour removal and control of suspended solids and turbidity
- A tertiary clarifier is used to clarify the water which is sent to a dual media filter and activated carbon filter

2.3.1 Dual media filter

A sand-anthracite filter or dual media filter/multi-media filter is primarily used for the removal of turbidity and suspended solids as low as 10-20 microns. Dual media filters provide very efficient particle removal under the conditions of a high filtration rate. Inside a sand-anthracite filter is a layered bed of filter media [1]. Sand is used to remove the suspended particles and anthracite is used to removing the odor and color from the effluent. Gravels and pebbles are provided for supporting both the media. To maintain the filtration efficiency, periodically an automatic back-washing technique is used.

2.3.2 Activated carbon filter

Here, the principle of adsorption is used. The filter medium adsorbs or reacts with pollutants in the effluent and filtered water is drained. The medium used is the natural material derived from coconut shell, lignite etc. the volatile organic matters, chlorine and disinfectants are removed from the effluent with the help of this filter. The removal of such impurities is important before the further filtration process like Reverse osmosis. The COD reduction upto the stage of an activated carbon filter is tabulated in below table 3.

Table 3 : COD& BOD reduction stage wise

Treatment	Reduction	BOD (mg/l)	Final BOD
Combined Effluent	---	800.00	800.00
Anaerobic Digester	45%	360.00	440.00
Primary Clarifier	20%	88.00	352.00
Biological Aeration	90%	316.80	35.20
Tertiary	20%	7.04	28.16
Dual Media Filter	5%	1.40	26.75
Activated Carbon Filter	5%	1.33	25.00

Treatment	Reduction	COD (mg/l)	Final COD
Combined Effluent	---	3000.00	3000.00
Anaerobic Digester	45%	1350.00	1650.00
Primary Clarifier	20%	330.00	1320.00
Biological Aeration	90%	1188.00	132.00
Tertiary	20%	26.40	105.60
Dual Media Filter	5%	5.28	100.32
Activated Carbon Filter	5%	5.01	95.31

2.4 Membrane filtration process:

After the secondary (biological) and tertiary (fine-tuning) treatment the effluent is subjected to the filtration system. The filtration system used here is micro-filtration, Reverse Osmosis (RO), and nano-filtration in series. Here, RO filtration is the heart of the system and to sustain its efficiency the pre-treatment of the effluent is a must and is done through the micro-filtration system. RO filtration separates the TDS or salts (both monovalent and divalent salts) from the effluent. Further to separate mono-valent salt (sodium chloride, sodium acetate etc) from divalent salt (Glauber salt) the technique of nano-filtration is used. Thus we can recover sodium chloride (in the form of brine solution) and Glauber salt solution (reject of nano-filtration) separately. The requirement, functions of each filtration system is described below.

2.4.1 Micro-filtration process:

- Used as a pretreatment for the reverse osmosis process.
- Operating pressure : 0.1-2.5 bar.
- Membrane pore size : 0.1-0.22 Micron
- Controlling parameters:Suspended solids and Turbidity < 1.
- Type of process:
 - Flushing :30 Sec,
 - Filtration :28 Min,
 - Backwash/Air Scouring : 60 Sec,
 - Valve actuation : 30 Sec.
- Input parameters required to be within the below limits
 - Total Suspended solids : < 10 ppm
 - Turbidity : < 10 NTU
 - Oil & Grease : < 2 ppm.
- Cleaning process:
 - Clean in Place – general : once in 15 days.
 - Chemical backwash : once in a day.
 - Cleaning Chemicals : Caustic 25%, Hypo 12%, Citric 2% and 98% Concentration.
- Microfiltration usually serves as a pre-treatment for other separation processes such as ultra-filtration, and a post-treatment for granular media filtration. The typical particle size used for microfiltration ranges from about 0.1 to 10 µm. In terms of approximate molecular weight, these membranes can separate macromolecules of molecular weights generally less than 100,000 g/mol. The filters used in the microfiltration process are specially designed to prevent particles such as sediment, algae, protozoa or large bacteria from passing through a specially designed filter. More microscopic, atomic or ionic materials such as water (H₂O), monovalent species such as Sodium (Na⁺) or Chloride (Cl⁻) ions, dissolved or natural organic matter, and small colloids and viruses will still be able to pass through the filter.
- The suspended liquid is passed through at a relatively high velocity of around 1–3 m/s and at low to moderate pressures (around 100-400 kPa) parallel or tangential to the semi-permeable membrane in a sheet or tubular form. A pump is commonly fitted onto the processing equipment to allow the liquid to pass through the membrane filter. There are also two pump configurations, either pressure-driven or vacuum. A differential or regular pressure gauge is commonly attached to measure the pressure drop between the outlet and inlet streams. The schematic diagram of the microfiltration process is as shown in the figure1 below.

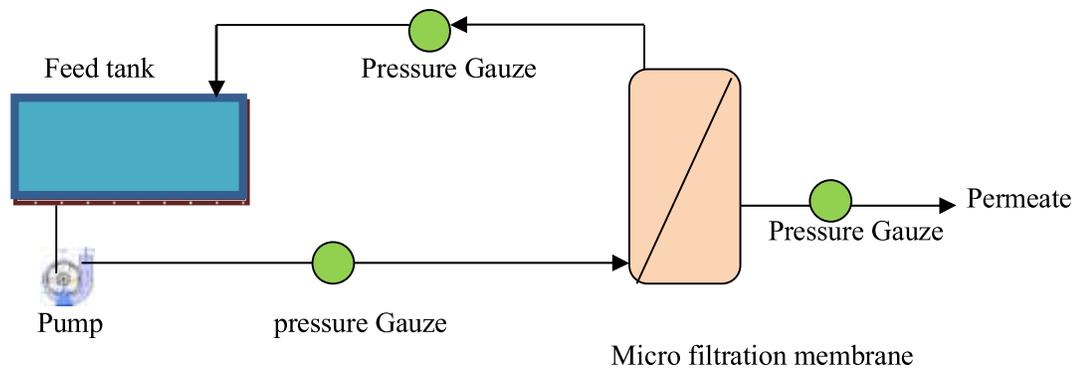


Figure 1 : Microfiltration process line diagram

2.4.2 Reverse osmosis process(RO)

2.4.2.1 RO process information

- Reverse Osmosis is the finest level of filtration available. The RO membrane acts as a barrier to all dissolved salts [2]. Reverse osmosis is a process that industry uses to clean water, whether for industrial process applications or to convert brackish water, to clean up wastewater or to recover salts from industrial processes.
- Reverse osmosis will not remove all contaminants from water as dissolved gases such as dissolved oxygen and carbon dioxide are not being removed. But reverse osmosis can be very effective at removing other products such as trihalomethanes (THM's), some pesticides, solvents and other volatile organic compounds (VOC's).
- In the reverse osmosis process cellophane-like membranes separate purified water from contaminated water. RO is when pressure is applied to the concentrated side of the membrane forcing purified water into the dilute side, the rejected impurities from the concentrated side being washed away in the reject water.
- RO can also act as an ultra-filter removing particles such as some micro-organisms that may be too large to pass through the pores of the membrane.
- The performance of a system depends on factors such as membrane type, flow control, feed water quality, temperature, and pressure.
- Also, only part of the water entering the unit is useable; this is called the % recovery. For example, the amount of treated water produced can decrease by about 1-2% for every 1 degree Celsius below the optimum temperature.
- Systems must be well maintained to ensure good performance with any fouling requiring cleaning maximizing the output of water.
- Biocides may be needed and the choice of biocide would depend on the membrane type, alternatively, other filters may be required to remove chlorine from water to protect the life of the membranes.
- To this end a good treatment regime is needed and

knowledge of the specific foulants so the optimum cleaning and maintenance chemicals can be chosen.

2.4.2.2 Reverse osmosis plant: Designed for 600 KL/day.

Here, 4 stages RO is designed to get maximum water recovery i.e. 91.5%. The inlet and outlet parameters in the RO plant in totality are given below

Table 4 :Inlet and outlet parameters in RO plant

Parameter	Treated water to RO	RO product water (average)	Unit
PH	6.5 – 7.5	6.0 – 7.0	-
Total suspended solids	< 1	Nil	Ppm
Total dissolved solids	6000	< 350	Ppm
Total Iron as Fe	< 0.1	Nil	Ppm
Calcium as CaCO3	75	< 10	Ppm
Total hardness as CaCO3	100	< 15	Ppm
Chlorides as Cl	4000	< 200	Ppm
Sulphates	1000	< 50	ppm
Total silica as SiO2	< 20	< 5	Ppm
Oil & Grease	Nil	Nil	Ppm
Organics	Nil	Nil	Ppm
Colour	Colourless	Nil	Ppm
Turbidity	< 1	Nil	Ppm
Heavy metals	Nil	Nil	Ppm

Table 5 : The stage-wise RO feed rate, TDS, % recovery and other parameters are given in the below table.

Particulars	unit	RO stage				Nano- filtration
		I	II	III	IV	
Designed capacity	KL/Day	600				75
Operating hours	Hours	20	17-18	13-14	17-18	20-21
Feed flow rate	KL/Day	30	12	8	4	3
Permeate flow rate	KL/Hr	19.5	6	2.8	1	1.5
Recovery	%	65	50	35	25	50
Reject flow rate	KL/Hr	10.5	6	5.2	3	1.5
Feed pressure required	PSI	450	600	800	900	600
Feed TDS	Ppm	6000	17000	33600	51300	68000
Permeate TDS	Ppm	< 300	< 400	< 400	< 400	60000
Reject TDS	ppm	17000	33600	51300	68000	73000

Thus, the overall recovery of good water (TDS < 400 ppm) is 91.5%

2.4.3 Nano-filtration (NF) plant

RO and NF are both membrane technologies that use a semi-permeable medium to remove certain ions and particles from a liquid stream, they can be distinguished based on the size of particulates that each can remove. Comparatively, RO and NF are capable of removing finer contaminants than micro-filtration and ultra filtration [3]. Nano-filtration membrane has pore sizes from 1-10 nano-meters, smaller than that used in microfiltration and ultra-filtration, but just larger than that in reverse osmosis. Membranes used are predominantly created from polymer thin films. The original uses for nano-filtration were water treatment and in particular water softening

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1. <https://www.chemtronicsindia.com/dual-media-filter.htm>
2. Handbook on ETP, water recycling& sustainable technology by BTRA Mumbai page no. 53
- 3 A fundamental guide to industrial reverse osmosis and nano-filtration an E-book by Samco technologies- chapter 1, page no. 6 (<https://www.samcotech.com/reverse-osmosis-nanofiltration>)

The reject effluent quantity of 68-70 KL from 4th stage RO, which is having a TDS value of 51000-52000 ppm is fed to a nano-filtration system to separate and recover sodium chloride, sodium acetate and any other mono-valent salt brine solution from Glauber salt solution. The plant running parameters are shown in the above table along with RO parameters. From the 70 KL RO reject is fed to using the nano-filtration and the permeate 35 KL with 60 grams/lit salt in it called as brine solution is gain used in the dyeing department with required top of the fresh salt.

In next part III, we will be discussing the multiple effect evaporation, mass balance, salt recovery, Sludge generation and disposal part so that complete ZLD is achieved

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Surface Modified, Rare Earth Mordanted Cotton, Dyed with Eupatorium Extract

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Abstract

Eupatorium dye extract has been used for dyeing cotton using rare earth (RE) metal mordants such as Cerrous sulphate, Lanthanum chloride and Yttrium chloride and compared with conventional metal mordant Alums. The metal salts were used for premordanting. The cotton surface was modified before premordanting with an auxiliary. The enhancement in CIELab, K/S and E values were found to be very good. The betterment in dye exhaustion along with improvement in the wash and light fastnesses with the auxiliary mediated surface-modified cotton using rare earth salts as mordants has been highlighted in this paper.

Keywords

Eupatorium; Cotton fabric; Rare earth metal mordant; Auxiliary; K/S value

Introduction:

Eupatorium species have been used for the natural dyeing of natural fabric by several workers. *Eupatorium adenophorum* was used as a very good green colour source for dyeing of silk yarn with excellent fastness properties. Four mordants were used in 1-5 % with three methods of mordanting. Results show excellent fastness to light and outstanding fastness to washing. Little noticeable staining and colour change was found for dry and wet crocking samples [1].

Eupatorium odoratum was used as a source of natural dye for cotton dyeing. It was found that cotton yarn, pretreated with a chitosan solution (with and without a crosslinking glyoxal solution), followed by dyeing with *E. odoratum* extract in the presence of alum, provided a better depth of shade (K/S) and also gave better fastness to light and washing than the untreated cotton yarn. Pretreated cotton yarn with the biomordant solution from *Memecylonscutellatum* leaves gave relatively poor light and wash fastness of the resultant dyed cotton than in the presence of alum [2].

Dyeing of protein fabrics exploring locally available weed plants such as *Lantana Camara*, *Solanum nigrum*, *Eupatorium adenophorum*, *Tridax procumbens*, *Parthenium hysterophorus*, *Sida acuta*, *Ipomea cairica*, *Rumex*

nepalensis, *Eclipta prostrate*, *Girardinia diversifolia*, *Erigeron bellidioides*, *Bidens pilosa* and *Athadavasica* which were easily available in hilly as well as Tarai regions of Uttarakhand was attempted for natural dyeing. Dyeing of wool and silk fabrics were carried out. It was found that various plants like *E. adenophorum*, *R. nepalensis* and *B. pilosa* gave acceptable colour on silk and wool fabric. Apart from the weed plants which can be used for textile dyeing they can also provide a scope for the management of weed plants along with the safety of other useful indigenous plants [3].

The yellow flavonoid constituents from *E. odoratum* also have potential use as dyestuffs for textile dyeing to retain a yellow colour in the dyed cotton using alum as a safe mordant [4]. Chaiwong et al. [5] attempted dyeing of cotton with an extract from *E. odoratum* (Saab Suea) in the presence of iron(II) sulphate, which gave a green to black shade.

The significance and importance of Eupatorium dye can be well understood as three Chinese patents and one Indian patent have been published in recent years. Some are with different species of Eupatorium-A kind of Eupatorium cannabinum L. (*Eupatorium coelestinum* L.) dye liquor and preparation method thereof and purposes (Patent no CN106118126A) [6]. The second is based on the Eupatorium adenophorum dye and dyeing process thereof (Patent no CN101284951B) [7]. The third one is a kind of preparation

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method of plant dyeing liquid(Patent no CN106967305A) [8]. An Indian patent- a process of preparation of Herbal dye suitable for cotton and woollen fabrics Indian patent no 242803 [9].

The reason for using Eupatorium as a dye source is twofold- firstly it is freely available and secondly has good colorant content. This makes the Eupatorium dye extract an ideal candidate for natural colourant.

Colorant present in the leaves of *Eupatorium adenophorum* are mainly Flavonoids such as Apigenin, Rhamnetin, Luteolin, Quercetin, Kaempferol, Tamrixetin, and Dihydrokaempferid[2]. Structurally they are best suited for metal mordanting as shown in the figure-1 given below.

Material and Methods:

Material: The cotton fabric was purchased from Sanjay Shah and Associates RFD of GSM-130 gm/m², Eupatorium dye was bought from Avani Earthcrafts, Uttrakhand, Auxiliary was supplied by Dyetex, Mumbai. Rare earth salts (RE)- cerous sulphate, lanthanum chloride and yttrium chloride were purchased from IREL, Cochin. All other reagents were purchased from a local vendor.

Measurement of reflectance (%) & colour strength

(K/S) Reflectance (%) of the dyed fabric samples were measured by using a Premier Colour scan spectrophotometer. As the strength of any dye is related to its absorption property, so by using Kubelka–Munk formula,

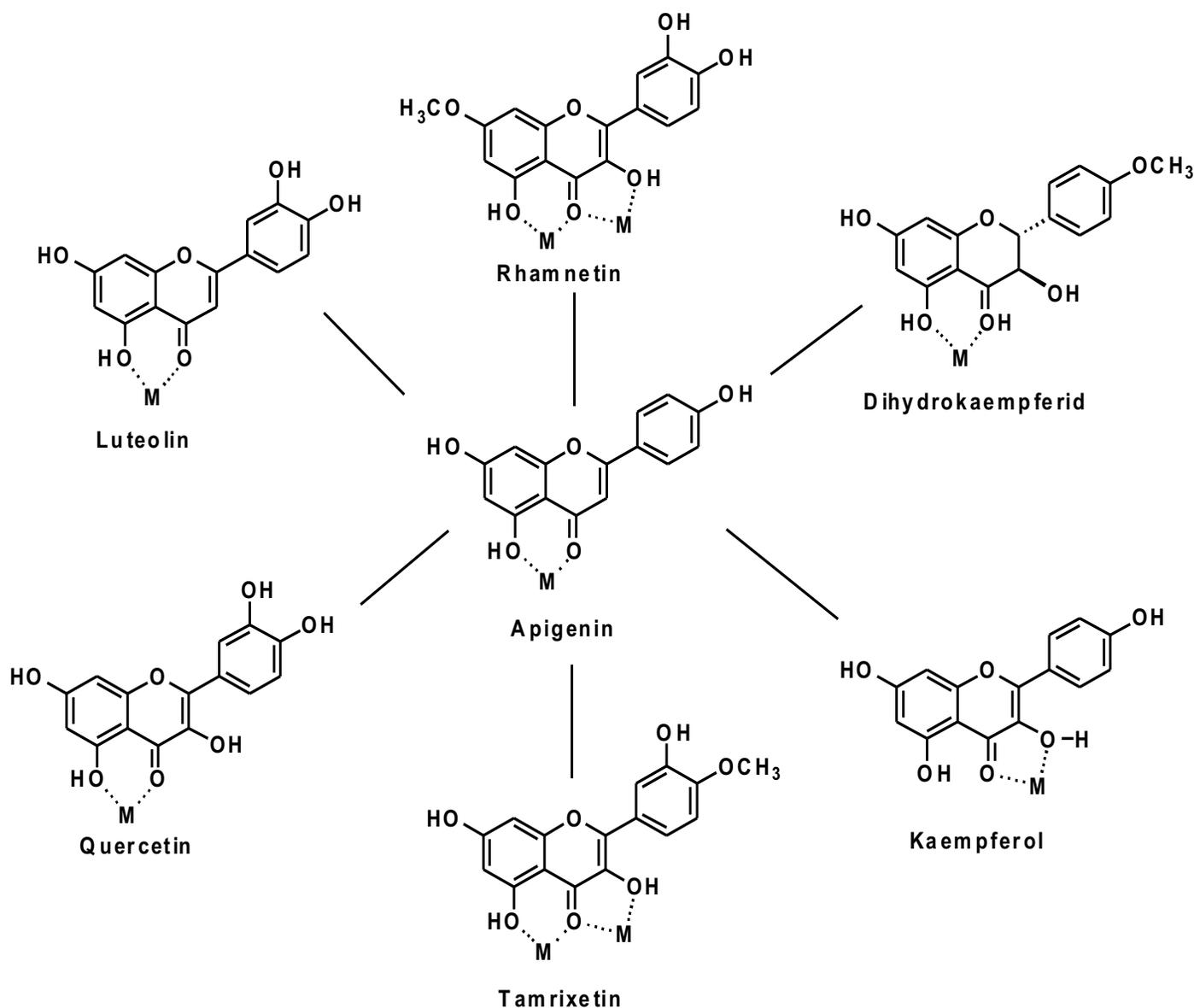


Figure 2 : Flavonoid Components of Eupatorium

the following relation between reflectance and absorbance can be derived:

$$K/S = \left[\frac{(1-R)^2}{2R} \right]$$

Where R is the reflectance, K is absorbance and S is the scattering. By using the above equation colour strength of different dyed samples was measured.

Evaluation of Fastness Properties

Launder-o-meter for testing washing fastness and fadometer with xenon arc lamp for testing light fastness were used. The dyed samples were tested according to Indian standard methods.

The specific tests were: colour fastness for light, IS-2454-85. The washing fastness of samples was evaluated by the standard test method, ISO: 105 C: 10:2006.

Surface modification of Cotton

The process of modifying cotton by developing an active site on its surface without affecting its bulk property can be easily done on the cotton fiber to increase dye-fiber interactions is thus, the best route to overcome the lack of affinity for cotton to natural dyes, so that it can be dyed without any additive. It was found that during the activation of cotton, etherification of primary hydroxyl groups on cellulose takes place [10,11]. The introduction of activated sites within the cellulose is the most expected technique to increase dye adsorption. Cationic sites can be introduced either by aminization or cationization. Cationization is one of the most important modifications for cellulose. The cationization is mainly carried out to improve affinity toward anionic substances, such as dyes in conventional textile processing and metal ions or unfixed dyes in effluent treatment. Cationic modification is the method that has been employed to change the surface charge of cellulosic fibers [12,13]. The common cationizing methods of cellulosic materials are by three ways: 1) a direct cationization of cellulose using a chemical compound with suitable functional groups that react with cellulose hydroxyl groups. 2) Through a binding agent such as DMDHEU which reacts both with cellulose hydroxyl and the functional group of cationic agents. This process is mainly used for textile application since the common textile pad-dry-cure process can be employed. 3) Through graft polymerization to introduce monomeric or polymeric cationizing agents within the cellulose. These methods are not commercially viable [12].

By the introduction of cationic groups into cotton fibers, the dye adherence for cotton can be significantly improved. The ionic attractions between cationized cotton and reactive dyes can result in increased dye uptake, reduced or no electrolyte used, less dye washing off and less water and energy consumption. The environmental problems caused by dye and salt in the effluent can be potentially mitigated by cationization pretreatment of cotton [14,15]. Instead of focusing on the synthesis of new dyes and modification of the cotton dyeing process, much research has focused on the modification of cotton at the molecular level to obtain the

desired dyeing performance and colorfastness properties with existing dyes. In all well-studied modifications of cotton, cationization is effective in increasing the dye uptake of anionic dyes.

Our attempt for using an auxiliary (PA) as a surface modifier before pre-mordanting with rare earth salts has been very encouraging in the case of dyeing cotton fabric with Eupatorium dye. The effect of auxiliary in facilitating dye diffusion into the fabric acting as a surface modifier and the role of rare earth salts- cerous sulphate, lanthanum chloride, and yttrium chloride in dye-binding have shown good dye adherence and colour depth.

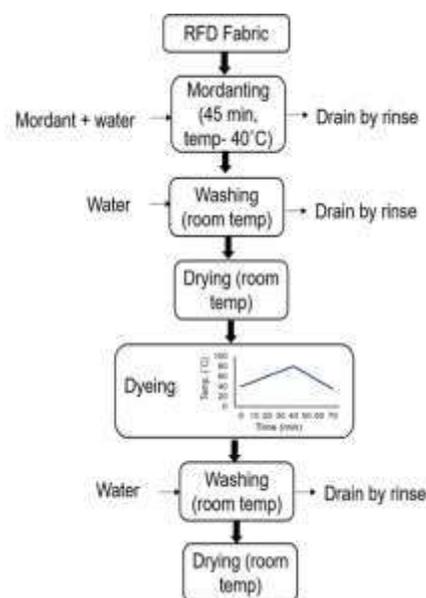


Figure 6: Mordant + Dyeing

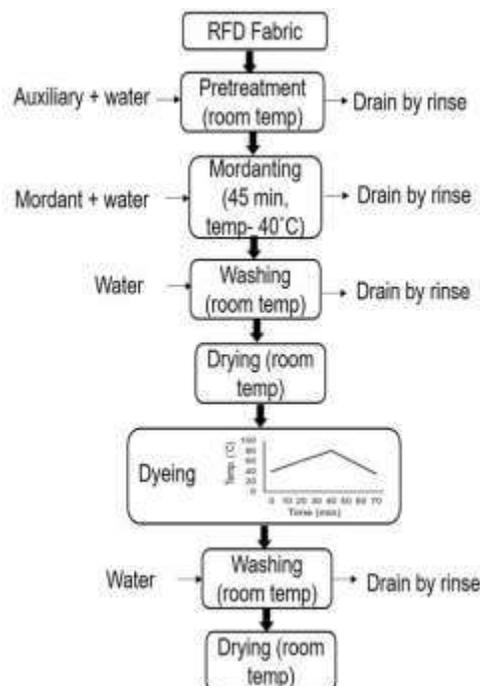


Figure 7: PA+Mordant + Dyeing

The dyeing sequence is shown in Figure 6 and 7.

As shown in figure 6 and 7, the dyeing procedure of cotton fabrics was followed in 03 main steps respectively -

1. Auxiliary pretreatment (PA)
2. Pre-mordanting with metal mordant salts
3. Dyeing with natural dye- Eupatorium

Auxiliary pretreatment- The process of pretreatment was carried out with 5% solution of the auxiliary by maintaining 1:30 MLR in a water bath. The process was carried out at room temp with gentle stirring continued for 45 minutes.

Pre-mordanting with metal mordant salts- Pre-mordanting was carried with 1% solution of metal mordant such as alum, cerous sulphate, lanthanum chloride and yttrium chloride salts by maintaining 1:30 MLR in a water bath. The pre-mordanting was carried out at 40°C with gentle stirring continued for 45 minutes.

Dyeing with natural dye- Eupatorium- Dyeing of cotton fabrics were carried in a water bath by maintaining the MLR 1:30, temp 35°-80°C for 70 minutes. After dyeing samples were washed with water and dried at room temperature.

Dyeing Procedure for cotton fabrics as shown in figure 6 and 7

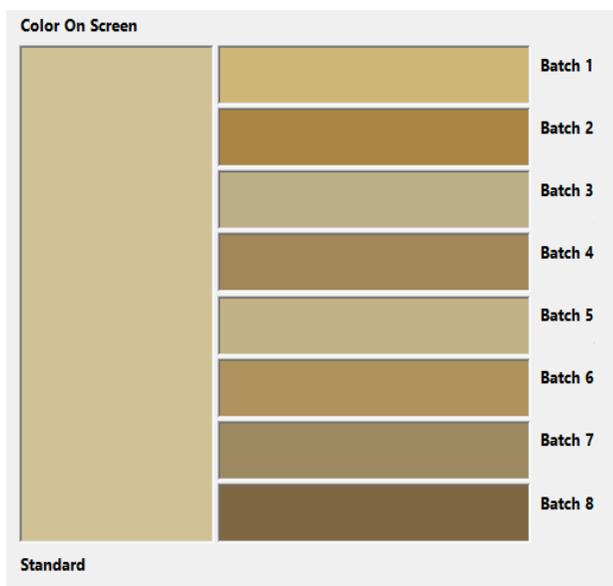


Figure - 8 Standard: Unmordanted control, Batch -1: Alum mordanted, Batch-2: Alum+ PA, Batch-3: Cerous sulphate, Batch-4: Cerous sulphate +PA, Batch-5: Lanthanum chloride, Batch-6: Lanthanum chloride +PA, Batch-7: Yttrium chloride, Batch-8 Yttrium chloride +PA

It is very apparent from the figure-8 that the colour obtained after dyeing with only eupatorium extract and alum, cerous sulphate, lanthanum chloride and yttrium chloride mordanted (batch 1, 3, 5 and 7) are much lighter in shade and colour depth than auxiliary pretreated, metal mordanted and

finally dyed cotton samples (batch 2, 4, 6 and 8). The same is reflected in the CIElab values and K/S values of the different batches as shown in table -1.

Measurement of K/S value is an easy and simple operation that can be done on any colour scanning machine. K/S value can indicate the depth of the colour of the dyed fabric surface. It has been shown by Lui and co-workers that dyeing with Reactive Blue B-2GLN has shown a direct relationship between K/S value and the fixing rate with the reactive dyes on cotton fabric. The results show that the K/S value of the fabric has a good linear relationship to the reactive dyes fixation rate [16].

The Eupatorium dyeing result shown in table-1 indicates that the auxiliary pretreated cotton has shown significantly increased dye uptake. This is because of cellulose chains having strong ionic attractions between the natural dye-Eupatorium and the surface-modified cotton fabric which helped better dye absorption onto the fabric surface. The kinetics of conventional dyeing has mainly 4 stages: dye diffusion in bath, adsorption, diffusion into the fabric, and chemical reaction. Due to significantly improved affinity for the dye and increased dye concentration on the fabric surface, the auxiliary pretreated cotton has an increased concentration gradient for the diffusion of the dye into the fabric. The increase in concentration gradient is expected to accelerate the dye diffusion. The remarkable role of the auxiliary in enhancing the K/S and ΔE values can be seen in table -1 (serial nos 2, 4, 6 and 8). Among the three rare earth salts used the highest value for K/S and ΔE were obtained for the yttrium chloride treated sample. The overall best result was observed for the PA+yttrium chloride treated sample.

Fastness properties shown in table-2, show that alone premordanting with rare earth salt did not show much change in the wash and light fastnesses, however when the cotton fabric was pretreated with auxiliary there was a considerable enhancement in fastness values as can be seen in serial nos. 2, 4, 6 and 8. Although cerous sulphate and yttrium chloride showed equal wash fastness, light fastness for yttrium chloride treated fabric was best. Overall best results were obtained for PA+yttrium chloride treated sample.

Conclusion: Cotton dyeing with Eupatorium dye has been attempted by several researchers using different types of mordants and/or fixing agent which are either not safe chemicals or have to be used in fairly high quantities almost up to 5-6%. We have shown the use of rare earth salts in 1% can do the requisite chelation of the colourant. The RE salts are eco-friendly and are thus a safe alternative. The use of Rare earth salt premordanting has enhanced dye adherence and offered remarkable improvement in the fastness properties of the dyed swatches. Thus it can be concluded that pretreatment with unique auxiliary coupled with pre mordanting with RE salt –Yttrium chloride has made a significant improvement in the fastness properties of Eupatorium dyed cotton sample utilizing its unique chelation property.

Table-1 CIELab Value of Eupatorium Dyed cotton

S.No	Name	K/S INT	K/S MAX	L*	a	b	C	H	dE*
Std	Control	7.624	1.639	75.20	-0.85	23.16	23.17	92.14	---
1	Alum	13.750	2.720	74.52	0.64	33.84	33.85	91.19	11.40
2	PA*+Alum	54.907	9.447	57.56	7.08	41.09	41.69	80.18	28.47
3	Cerous sulphate	11.822	1.989	71.74	-1.74	22.75	22.81	94.42	6.53
4	PA*+ C S	36.038	6.079	57.84	4.23	28.17	28.48	81.42	21.56
5	Lanthanum chloride	11.804	2.207	72.63	-0.06	24.27	24.27	90.19	5.73
6	PA* + L C	30.783	5.570	61.88	4.02	31.77	32.02	82.75	19.08
7	Yttrium chloride	31.490	4.487	57.97	1.21	24.76	24.79	87.16	20.39
8	PA* + Y C	72.791	9.510	45.14	4.46	23.93	24.34	79.39	33.49

*PA is the Auxiliary, used as a pretreatment before mordanting

Table-2 Fastness properties of Eupatorium dyed cotton fabrics

S.No	Name	Washing fastness	Lightfastness
Std	Control	3-4	3
1	Alum	3-4	3
2	PA*+Alum	4-5	5
3	Cerous sulphate	3	3
4	PA*+ C S	4-5	4-5
5	Lanthanum chloride	3	3-4
6	PA* + L C	3-4	4-5
7	Yttrium chloride	4	5
8	PA* + Y C	4-5	5-6

Acknowledgment: The authors express their thanks to the funding agency Indian Rare Earth Limited for supporting this research work.

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Study of Banned Amines Recovery

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Abstract

This study describes the Spike Recovery of all 24 Banned Amines by following the European standard EN 14362. The work is carried out by directly using the standard Banned Amines and not with the azo colorants. We can see the poor recovery of some of the amines due to their poor stability and their further reaction in an extraction process. To prevent the further reaction of standard Banned Amines to some other form of amines or compounds, it is necessary to skip the Reductive Cleavage step for the recovery study of some of the amines namely o-aminoazotoluene, 2-amino-4-nitrotoluene and 4-aminoazobenzene. Due to the less recovery of 2,4-diaminoanisole and 2,4-diaminotoluene, it is necessary to apply a correction factor of recovery when it is detected in any sample.

Keywords

Banned Amines, Azo dyes, Spike Recovery

1.0 Introduction

Azo dyes are organic compounds bearing the functional group $R-N=N-R'$, in which R and R' are usually aryls. They are a commercially important family of azo compounds, i.e. compounds containing the linkage C-N=N-C. Azo dyes are widely used to treat textiles, leather articles, and some foods.

Azo dyes derived from benzidine are carcinogens; exposure to them has classically been associated with bladder cancer. Accordingly, the production of benzidine azo dyes was discontinued in the 1980s in many western countries. Certain azo dyes degrade under reductive conditions to release any of a group of defined aromatic amines. Consumer goods that contain listed aromatic amines originating from azo dyes were prohibited from manufacture and sale in European Union countries in September 2003 due to their carcinogenic effect.

European standard EN 14362 is a method for 'Determination of Banned Amines Derived from Colorants'. Though it is a standard method, for any residual level analysis, it is a good laboratory practice to derive –

- Instrument Detection Limit (IDL)
- Method Detection Limit (MDL)

- Repeatability
- Reproducibility and
- Measurement Uncertainty (MU).

With the help of this data, we can ascertain the skill of the staff and so the competency of the laboratory. All this data can be derived either by Spike Recovery of the standard banned amines or by the re-testing of the reference samples. By this exercise, data for a few banned amines can be gathered. As there are 24 Banned Amines, it is very difficult to get the reference samples of azo colorants that can release all these amines.

To get more confidence to analyse an unknown sample of azo colorants, it is necessary to have Recovery data of all 24 Banned Amines. This study is to derive the recovery data of Banned Amines.

2.0 Standard Method: EN 14362-2009

Method for determination of certain aromatic amines derived from azo colourants.

3.0 Reagents and Materials

- RFD (Ready for Dyeing) cotton fabric
- 24 standard amines
- Citrate buffer

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- Sodium Dithionite
- Methanol
- t-butyl methyl ether
- Grade 3 water

4.0 Apparatus

- Water bath, Vacuum rotary evaporator, Pipette
- HPLC (High-Performance Liquid Chromatograph)
- GC-MS (Gas Chromatograph-Mass Selective Detector)

5.0 Standard Solution Preparation

1000 ppm(parts per million) solution of all 24 banned amines was prepared separately. Then the mixture of amines was prepared in three groups by considering the Retention Time of the amines in GC-MS and HPLC so that there should not be any overlap of more than 1 amine in a chromatograph.

Table 1: Mixture of amines Group 1

Sr. No.	Banned Amine
1	o-anisidine
2	4,4'-methylenedianiline
3	2,6-xylidine
4	4,4'-thiodianiline
5	4,4'-methylenedi-o-toluidine
6	2,2'-dichloro-4,4'-methylenedianiline
7	3,3'-dimethoxybenzidine
8	3,3'-dichlorobenzidine

Table 2: Mixture of amines Group 2

Sr. No.	Banned Amine
1	2,4-diaminotoluene
2	4,4'-oxydianiline
3	o-toluidine
4	p-chloroaniline
5	p-cresidine
6	4-chloro-o-toluidine
7	4-aminobiphenyl
8	2,4,5-trimethylaniline
9	o-aminoazotoluene

Table 3: Mixture of amines Group 3

Sr. No.	Banned Amine
1	2,4-diaminoanisole
2	2-amino-4-nitrotoluene
3	2,4-xylidine
4	2-naphthylamine
5	Benzidine
6	3,3'-dimethylbenzidine
7	4-aminoazobenzene

From this mixture of standard amines three different concentrations of 50, 100, and 200 ppm(parts per million) were made. These solutions were then used for the study of spike recovery by following the standard method EN-14362:2009 (2.0).

Standard Operating Procedure (SOP)

- I. Spike the Prepared standard solution on 1 gm small pieces of 100% cotton Ready for Dyeing (RFD) fabric.
- II. Reaction with citrate buffer at 70°C for 30 minutes followed by reaction with Sodium Dithionite at 70°C for 30 minutes.
- III. Cool at room temperature followed by solvent extraction and concentration of Amine.

Analysis of the final prepared solutions by GC-MS for the identification of amines, followed by Quantitative Analysis by HPLC.

6.0 Results and Discussion

Table 4: Recovery Data

Sr. No.	Banned Amine	% Recovery		
		50 ppm	100 ppm	200 ppm
1	4-aminobiphenyl	82.0	80.1	73.9
2	Benzidine	66.8	66.8	60.0
3	4-chloro-o-toluidine	86.8	81.4	70.7
4	2-naphthylamine	85.3	*	*
5	o-aminoazotoluene	81.5	*	*
6	2-amino-4-nitrotoluene	97.2	*	*
7	p-chloroaniline	82.8	78.0	67.8
8	2,4-diaminoanisole	18.3	20.3	18.9
9	4,4'-methylenedianiline	88.8	83.9	76.5
10	3,3'-dichlorobenzidine	59.1	57.5	63.4

Sr. No.	Banned Amine	% Recovery		
		50 ppm	100 ppm	200 ppm
11	3,3'-dimethoxy-benzidine	91.1	86.9	79.2
12	3,3'-dimethyl-benzidine	87.9	86.7	81.5
13	4,4'-methylene-di-o-toluidine	95.3	90.2	82.5
14	p-cresidine	87.1	81.9	71.2
15	2,2'-dichloro-4,4'-methylene-dianiline	66.7	66.9	66.9
16	4,4'-oxydianiline	83.7	81.8	74.0
17	4,4'-thiodianiline	67.4	67.6	59.7
18	o-toluidine	100.0	100.0	94.0
19	2,4-diaminotoluene	35.4	38.8	39.0
20	2,4,5-trimethylaniline	70.0	nd	Nd
21	o-anisidine	82.8	82.0	73.0
22	p-aminoazobenzene	78.4	*	*
23	2,4-xylydine	73.2	75.9	70.6
24	2,6-xylydine	93.2	87.6	80.3

*Data available only for 50 ppm

Based on the recovery data, the following is the inference of findings:

Three of the banned amines namely o-aminoazotoluene, 2-amino-4-nitrotoluene, and p-aminoazobenzene were not getting recovered in the group analysis.

Hence the spike recovery study was separately carried out for each of these 3 amines by spiking the amine in citrate buffer instead of RFD fabric. After the GC-MS analysis, it was observed **only a sharp peak of 1,4-benzenediol (Figure 1 for o-aminoazotoluene and Figure 3 for p-aminoazobenzene)***. This may be due to the further reaction of the amine group into 1, 4-benzenediol after the reductive cleavage and solvent extraction steps.

The spike recovery study was again carried out by spiking the standard amine solution in water followed by solvent extraction. The prepared solutions of these amines were then injected in a GC-MS. It was observed that **along with the amine peak, there was also a sharp peak of 1,4-benzenediol (Figure 4 for o-aminoazotoluene and Figure 5 for p-aminoazobenzene)**. This may be because of the further reaction of the amines in a solvent extraction process.

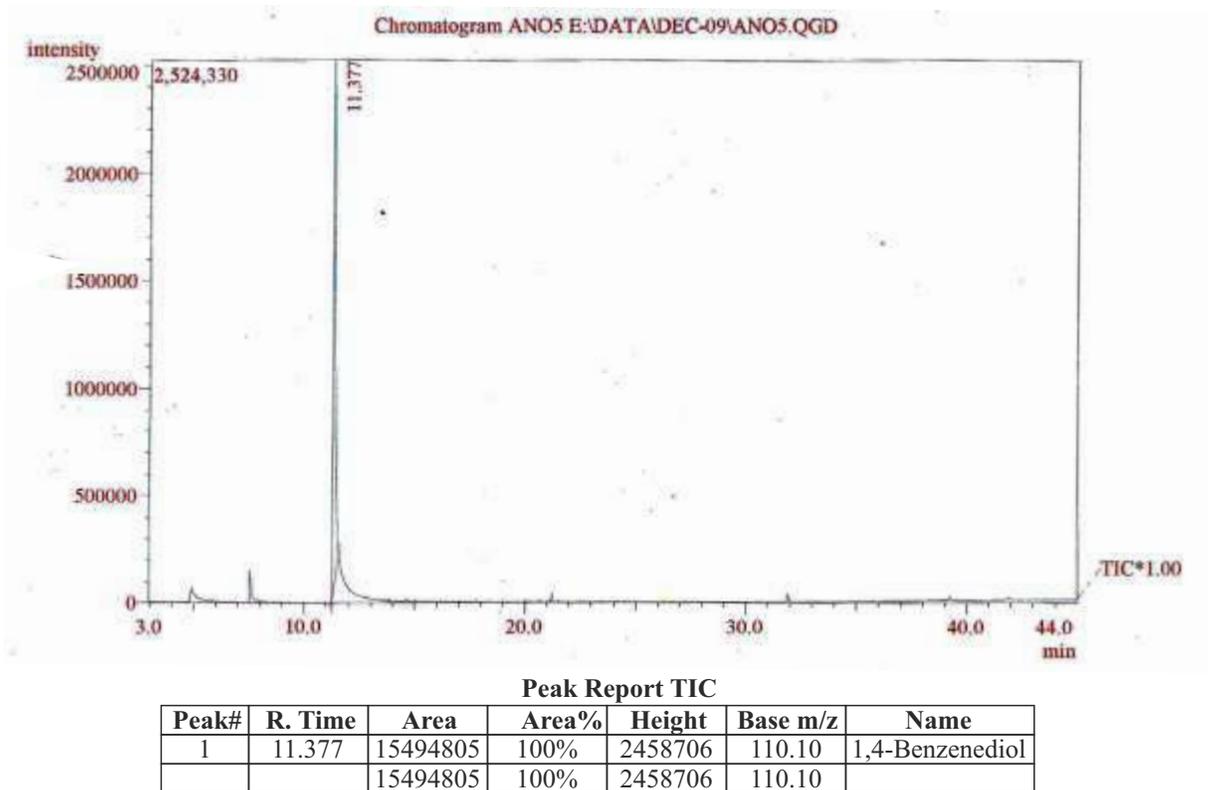
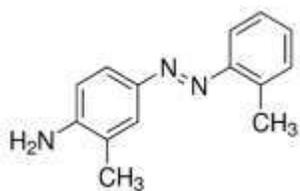
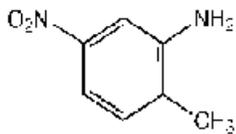


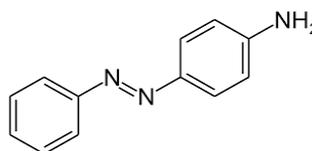
Figure 1: TIC of Spike Recovery Extract of o-aminoazotoluene (Spiking in Citrate buffer)



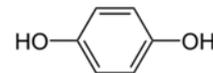
o-aminoazotoluene



2-amino-4-nitrotoluene



p-aminoazobenzene



1,4-benzenediol

Figure 2 : o-aminoazotoluene, 2-amino-4-nitrotoluene, p-aminoazobenzene and 1, 4-benzenediol

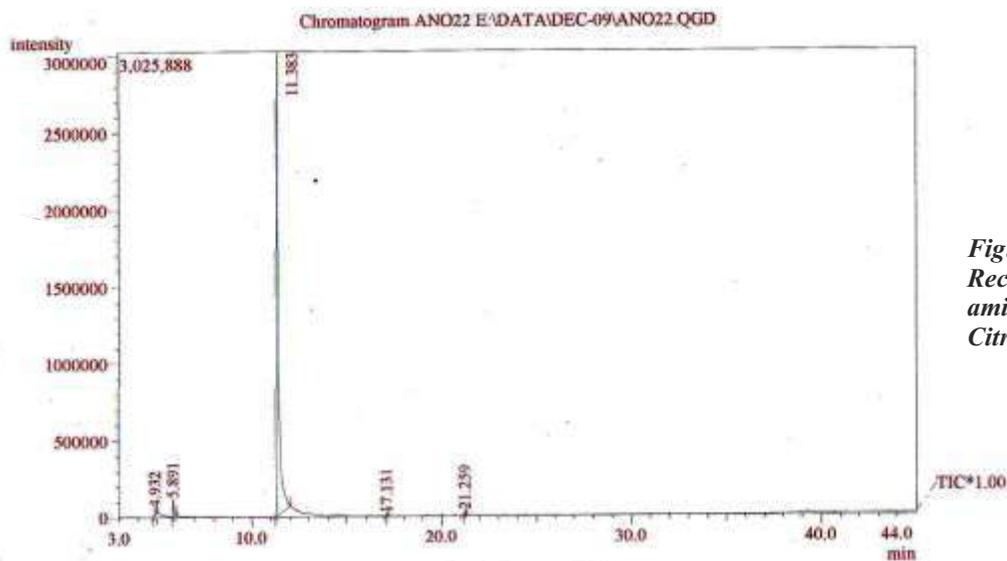


Figure 3: TIC of Spike Recovery Extract of p-aminoazobenzene (Spiking in Citrate buffer)

Peak Report TIC

Peak#	R. Time	Area	Area%	Height	Base m/z	Name
1	4.932	263286	1.03	47400	54.05	
2	5.891	430943	1.69	111774	93.10	
3	11.383	24712473	96.81	3011245	110.10	1,4-Benzenediol
4	17.131	33946	0.13	13247	73.10	
5	21.259	85981	0.34	31872	149.10	
		25526629	100%	3215538		

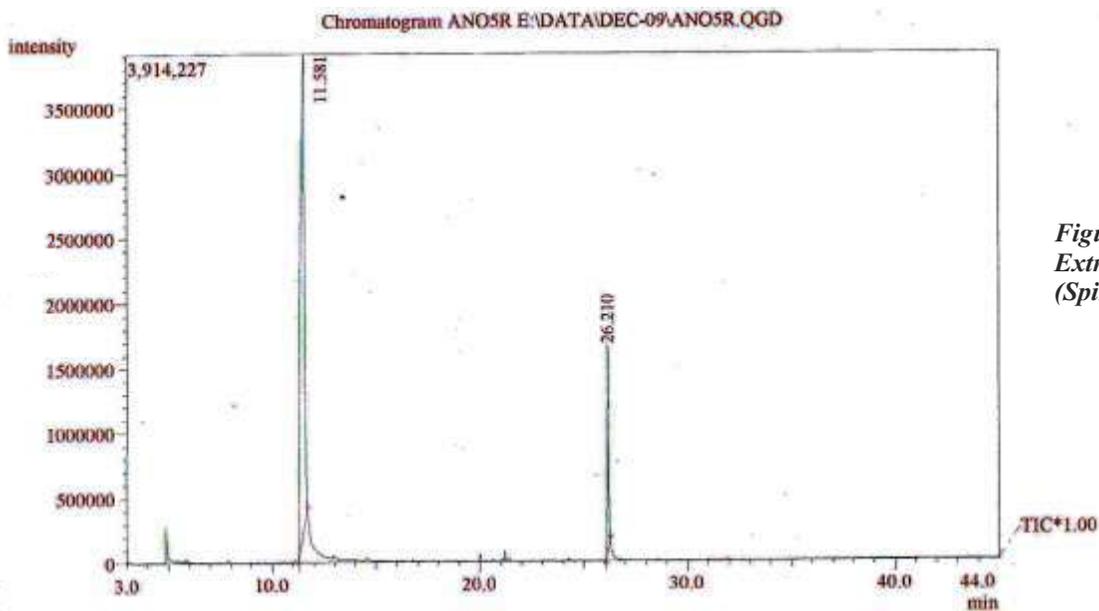
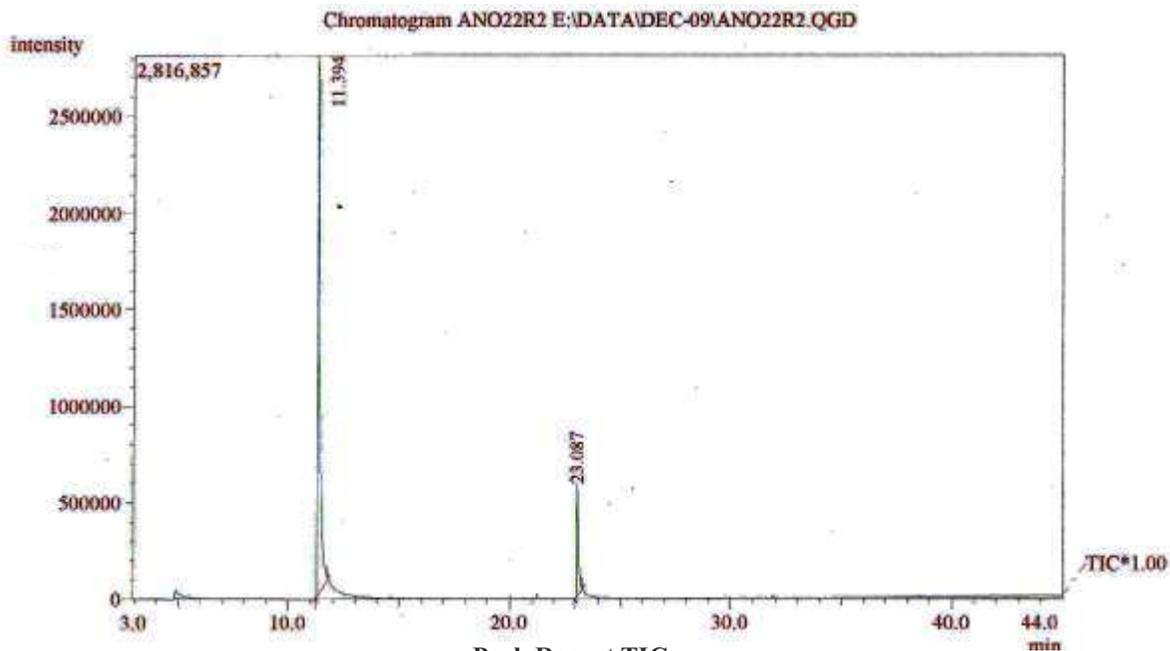


Figure 4: TIC of Spike Recovery Extract of o-aminoazotoluene (Spiking in water)

Peak Report TIC

Peak#	R. Time	Area	Area%	Height	Base m/z	Name
1	11.581	49954498	88.61	3643725	110.10	1,4-Benzenediol
2	26.210	6418556	11.39	1592464	106.15	o-Aminoazotoluene
		5673054	100.00	5236189		



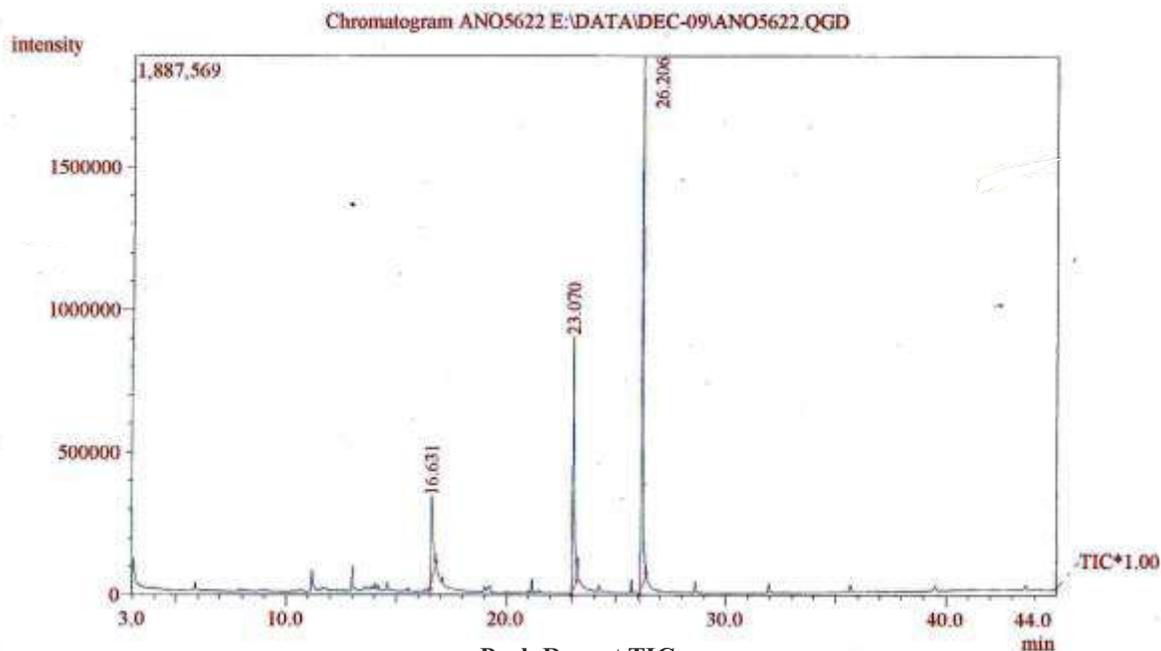
Peak Report TIC

Peak#	R. Time	Area	Area%	Height	Base m/z	Name
1	11.394	20757702	88.66	2792506	110.10	1,4-Benzenediol
2	23.087	3196323	13.34	578867	92.10	p-Aminoazotoluene
		23954025	100.00	3371373		

Figure 5: TIC of Spike Recovery Extract of p-aminoazobenzene (Spiking in water)

This was again confirmed by injecting the standard amine solution directly in GC-MS. TIC of this clearly shows a very sharp peak of these 3 amines (Figure 6). Hence, it was

confirmed that the 1,4-benzenediol peak was because of the further reaction of the amines in an extraction process.



Peak Report TIC

Peak#	R. Time	Area	Area%	Height	Base m/z	Name
1	16.631	1916462	13.38	300080	77.05	o-Toluidine, 5-nitro-
2	23.070	4173497	29.13	892170	92.10	p-Aminoazobenzene
3	26.206	8236797	57.49	1856342	106.10	o-Aminoazobenzene
		14326756	100.00	3048592		

Figure 6: TIC of Mixture of Standard amines o-aminoazotoluene, 2-amino-4-nitrotoluene, p-aminoazobenzene

Recovery of the 2 of the banned Amines namely 2,4-diaminoanisole and 2,4-diaminotoluene was less. Hence the spike recovery study was separately carried out by spiking the standard amine solution in citrate buffer instead of RFD cotton fabric. After the individual recovery study of these 2 amines, it was confirmed that there was not much improvement in the recovery.

7.0 Conclusion

In the recovery study, it was found that the stability of 3 of the Banned Amines namely o-aminoazotoluene, 2-amino-4-nitrotoluene, and 4-aminoazobenzene is poor and generates 1,4-benzenediol in an extraction process.

Recovery of 2,4-diaminoanisole is less than 30 % and 2,4-diaminotoluene is less than 60 %. It is necessary to apply a

Abbreviations

RFD : Ready for Dyeing

TIC : Total Ion Chromatograph of GC-MS

Spike Recovery: Introducing the known quantity of reference material and finding out the quantity recovered after following the standard protocol.

GC-MS : Gas Chromatograph-Mass Selective Detector

HPLC : High-Performance Liquid Chromatograph

ppm : (parts per million)

References:

- 1)EN 14362:2009, Methods for determination of certain aromatic amines derived from azo colorants.
- 2)EN 14362:2012 Part 1 Methods for determination of certain aromatic amines derived from azo colorants
Detection of the use of certain azo colorants accessible with and without extracting the fibres.
- 3)EN 14362:2012 Part 3 Methods for determination of certain aromatic amines derived from azo colorants.
Methods for determination of certain aromatic amines derived from azo colorants which may release 4-aminoazobenzene.

correction factor of recovery when it is detected in an unknown sample.

Part 3 was added in EN 14362 in 2012 as a special procedure for azo colorants that can form p-aminoazobenzene. Under the conditions of EN 14362-1, it generates the amines Aniline and 1,4-phenylenediamine.

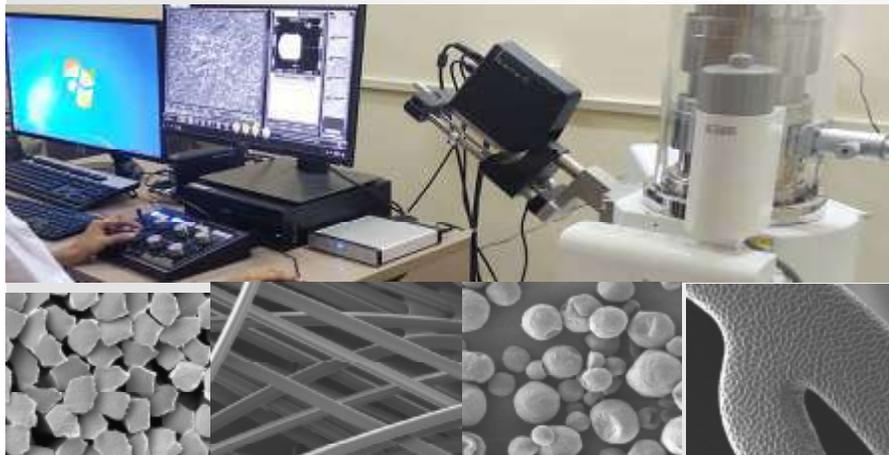
Under the conditions of EN 14362-1, the banned amines o-aminoazotoluene, 2-amino-4-nitrotoluene and p-aminoazobenzene generate 1,4-benzenediol.

Like part 3 for p-aminoazobenzene, it is necessary to study the azo colorants that can form o-aminoazotoluene and 2-amino-4-nitrotoluene.

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Adhesion Studies of Atmospheric Pressure Plasma Treated Nylon66 Fabrics with Polyurethane

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Abstract

Plasma treatment of nylon fabric was carried out with aim of improving the adhesion properties with polyurethane polymer. The effect of the plasma parameters vis. treatment time and power on adhesion improvement were examined. The force required to separate the coating from the fabric was measured as a peel-off strength of the coated fabric. Significant improvement in the adhesion of the plasma-treated sample was observed as compared to the untreated sample. Surface wettability after plasma treatment was studied by wicking measurements. A change in surface morphology was analysed using a scanning electron microscope (SEM). SEM results reveal the surface roughening after plasma exposure. The mechanical property was also studied by tensile strength measurements.

Keywords

Adhesion, Coating, Plasma treatment, Nylon 66, Polyurethane

1.0 Introduction:

Synthetic fibres are widely used in technical textiles and home furnishings due to their good physical and chemical properties. However, these fibres are hydrophobic due to the lack of polar functional groups. The hydrophobic nature of such fabrics limits their applications. Textile coatings are widely used in everyday life in different areas. The purpose of the coating is to provide its carrier material with specific functional properties for suitable application. The surface of the synthetic fibre is generally inert, making the fibre difficult to wet and hard to chemically bond to coating material, as a result, the adhesion between the fibre and coating material is inferior [1-4].

Adhesion is the tendency of dissimilar particles or surfaces to cling to one another. To ensure the maximum adhesion between polymeric material and coating; they must be compatible with each other in terms of hydrophilic and hydrophobic nature. An incompatibility between the polymeric material and the coating substance leads to poor interphase or adhesion which may limit its application. To improve adhesion to coatings, fibres are usually subjected to controlled surface treatments by wet chemical methods. However, chemical modifications have some disadvantages. For example, conventional methods are time-consuming and in most cases, are accompanied by a decrease in fibre

strength. Moreover, these conventional treatments can also lead to environmental pollution. Therefore, we propose to explore plasma techniques. Surface modification of textile fibres by cold plasma is simple and cost-effective. It produces no pollution and is becoming increasingly popular [5-11].

In this work, we have modified nylon 66 fabric using atmospheric pressure plasma generated from helium and helium mixed oxygen gases. The effect of plasma surface modification on the adhesion with polyurethane is reported in this paper.

2.0 Experimental

2.1. Materials and methods

A plain woven nylon 66 fabric with an area weight of 90 g/m², warp and weft density 70 and with 150 denier yarn fabric was plasma treated on an atmospheric pressure plasma reactor. Optimisation of the plasma process parameters was carried out by varying the plasma power and treatment time. Three different levels of plasma power and three different plasma exposure times were studied at a fixed distance between the electrodes at 0.5mm. Helium gas was used for the generation of plasma. Plasma treated fabrics were coated with polyurethane (commercial name: TUBICOAT MP SP) with a knife over roller coating method using a hand coating

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machine. The distance between the knife and roller was set at 3mm to apply the uniform coating. Drying and curing of the coated sample were carried out at 80°C for 10min and 140°C for 10 min respectively.

3.0 Characterization techniques

3.1 Uniformity of the coating

The uniformity of applied coating was studied by the thickness of and GSM of the coated fabric. The thickness of the uncoated and PU coated samples were tested as per ASTM D-1777-96 (2015) standard test method at 20 kPa pressure. Ten readings were taken randomly all over the fabric as directed in the test method and considered as the final thickness of the fabric. Similarly, Weight per square meter was measured as per ASTM D 3776-2013 standard. An average of five readings is considered as GSM of the fabric.

3.2 Wettability of plasma-treated samples

Change in hydrophilic and hydrophobic nature of nylon 66 fabric after plasma treatment was studied by the wicking height measurement. The rate of vertical capillary rise on plasma-treated samples was measured using the method described in ISO 9073-6; 2000 (E). Test specimens were suspended vertically in the liquid and checked for the increase in the capillary height at predetermined time intervals up to 20 min.

3.3 Adhesion strength test

T-peel off test was employed to measure the adhesion strength of coated fabrics according to the IS 7016 part 5-2011 test standard. Tinius Olsen, peel bond tester was used to measure the peel-off strength. Five different measurements were performed and the average value is considered as the bonding strength of the coated fabric.

3.4 Surface morphology by SEM

Surface topographical modifications in the nylon 66 samples before and after plasma treatment were investigated by scanning electron microscopy (SEM) on the JEOL SEM model JSM 5400 (Tokyo, Japan).

3.5 Mechanical properties

Tensile strength of the untreated and plasma-treated samples was carried out on pyramid tensile testing machine model Tinius Olsen H50KL Aimil. ASTM D 5035 -2015 standard test method was used. The average of the five test specimen was considered as the tensile strength of the fabric.

4.0 Results and Discussions

4.1 Uniformity of the coating

Table 1 gives the results of thickness and GSM of untreated and plasma-treated coated nylon 66 samples.

After coating thickness was increased by 0.29 ± 0.01 mm and GSM was increased by 376 ± 1 GSM. As per the standards 0.2mm and 5% variation in thickness and GSM is

Table 1: Thickness and GSM of plasma treated and coated nylon 66 fabric

Sr. No.	Sample name	Thickness in mm	GSM
1	Nylon fabric without coating	0.28	90
2	Untreated coated	0.57	467
3	Plasma treated 1.5kW/ 15 Sec	0.59	466
4	Plasma treated 1.5kW/ 30 Sec	0.59	466
5	Plasma treated 1.5kW/ 45 Sec	0.57	465
6	Plasma treated 2.5kW/ 15 Sec	0.58	465
7	Plasma treated 2.5kW/ 30 Sec	0.57	465
8	Plasma treated 2.5kW/ 45 Sec	0.59	467
9	Plasma treated 3.5kW/ 15 Sec	0.59	466
10	Plasma treated 3.5kW/ 30 Sec	0.58	467
11	Plasma treated 3.5kW/ 45 Sec	0.59	466

allowed respectively. In our case, the variation in thickness and GSM is well within the limits. Therefore, it can be said that the coating applied on the fabric is uniform all over the surface of nylon fabric.

4.2 Wettability studies on plasma-treated nylon fabric

Measurement of wicking height gives a good idea about the hydrophilic and hydrophobic nature of the textile fabric. The height of capillary rise was recorded for different wicking durations up to 20 min for plasma-treated and untreated nylon fabrics. The value of capillary height recorded for a predetermined time for both untreated and plasma-treated samples is plotted against the wicking time in figure 1.

It can be seen from figure 1, that all plasma-treated samples exhibit more wicking height as compared to untreated nylon sample. Further, as the plasma power increased from 1.5kW to 2.5kW wicking of the fabric improved. However, after 2.5kW of plasma power, there was no improvement in the wicking property of the plasma-treated samples. Hence it can be inferred that plasma power of 2.5kW is optimum to get maximum wettability. Similarly, the effect of plasma treatment time was also studied and it was found that the plasma exposure time of 30sec gives the maximum increase in wicking height. The increase in wicking height after plasma treatment can be attributed to a change in surface morphology which leads to an increase in the surface area and results in an increase in effective pore size and reduces the capillary pressure [12,13].

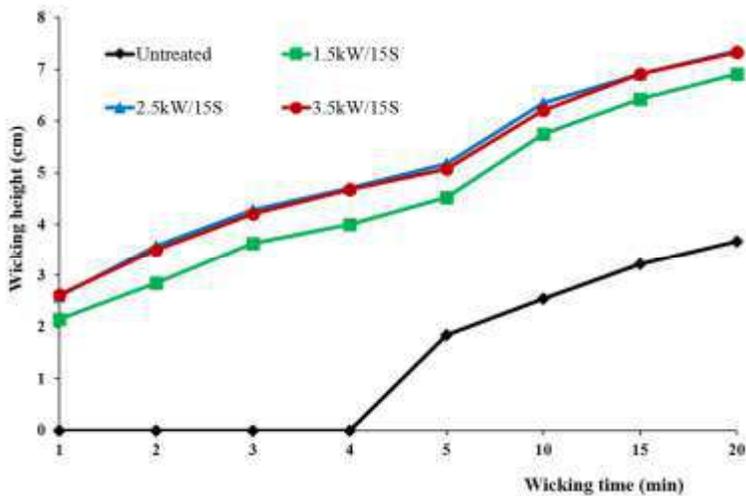


Figure 1: Wettability of plasma-treated nylon fabric, the effect of power

4.3 Adhesion studies of PU coated nylon fabric

Adhesion is the force required to separate the coating layer from the fabric. Adhesion force was measured as peel-off strength of the untreated and plasma-treated nylon coated samples and is given in Figure 2. The adhesion bond strength of plasma-treated coated samples showed a 20 -50 % increase compared to the untreated coated sample. It can be seen that with an increase in plasma power there is a gradual increase in adhesion strength. The highest peel bond strength value was obtained at a plasma power of 3.5kW. This suggests that by increasing the plasma power, the efficiency of plasma improves. Similarly, it was seen that with increasing the plasma exposure duration the adhesion strength was also improved. A similar trend concerning plasma exposure duration and power has also been reported by other researchers [14-16]. Improvement in adhesion strength after plasma treatment can be attributed to improved wicking which results in better spreading of the coating chemical and wetting of the nylon fabric which removed the weak boundary layer. Also, improved surface roughness proved better mechanical interlocking and improves the adhesion strength [17-19].

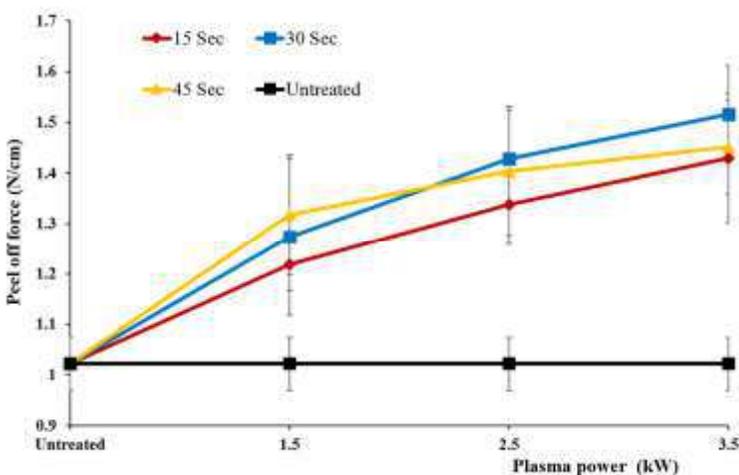


Figure 2: Peel off the strength of plasma-treated coated nylon samples

4.4 Surface morphology by SEM

Figure 3 shows SEM images of untreated and plasma-treated nylon samples. Morphological changes on the surface after helium plasma treatment can be observed (figure 3-B). The untreated (figure 3-A) nylon has a smooth and clean surface, while plasma-treated fabric shows a rougher surface. Helium plasma treatment etched the surfaces. Morphological alteration of the surface might lead to improved adhesion by providing better mechanical interlocking due to the roughening effect [20-22].

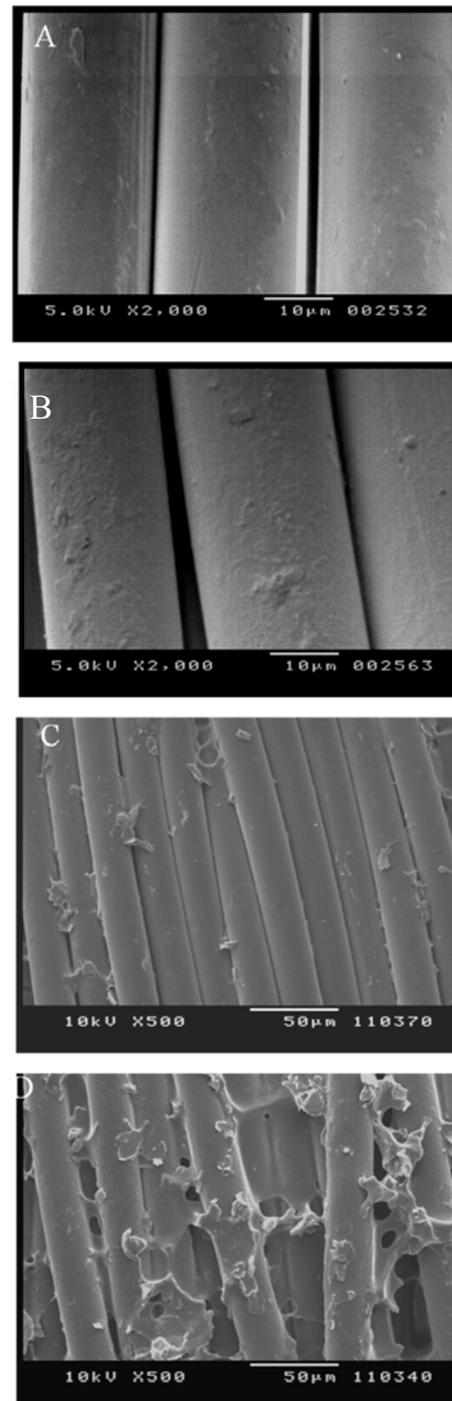


Figure 3: SEM photographs of nylon samples A- Untreated nylon, B- Plasma treated nylon, C- untreated after peel off and D- plasma-treated after peel off.

Figure 3-C, shows that for untreated samples after peel off testing, no coating material is left on the surface of fabric and coating is removed smoothly. The smooth surface after removal of coating may be due to no adhesion of the coating with fabric and hence the coating is removed smoothly. The bond failure occurs at the interface between the adhesive and nylon fabric. On the other, (figure 3-D) plasma-treated coated sample after peel off shows a considerable amount of PU film is left on the fabric surface this shows that there is better mechanical adhesion between the fabric and PU coating after plasma treatment. Hence adhesion strength is more after plasma treatment as shown in the above section. In this case, the adhesive itself breaks and particles of adhesive remain on the nylon surface. This type of bond failure is known as a cohesive failure.

4.5 Tensile properties of plasma-treated nylon fabrics

The tensile strength values of untreated nylon fabrics and after treatment with plasma are shown in Figure 4. The tensile strength of the untreated nylon (in warp direction) was 27.9N/mm. The tensile strength values after plasma treatment at power = 1.5, 2.5 and 3.5 for 15 sec treatment time were 29.04, 29.01 and 29.11N/mm, respectively. Figure 4 shows hardly any effect on the tensile properties after plasma treatment. Even, the percentage change in fabric tensile strength after plasma treatment is very small (up to 4%) in comparison with untreated nylon. Therefore, it may be inferred that the given plasma process parameters result in only a slight increase in the fabric tensile strength. During plasma, treatment etching occurs. The etching kind of the

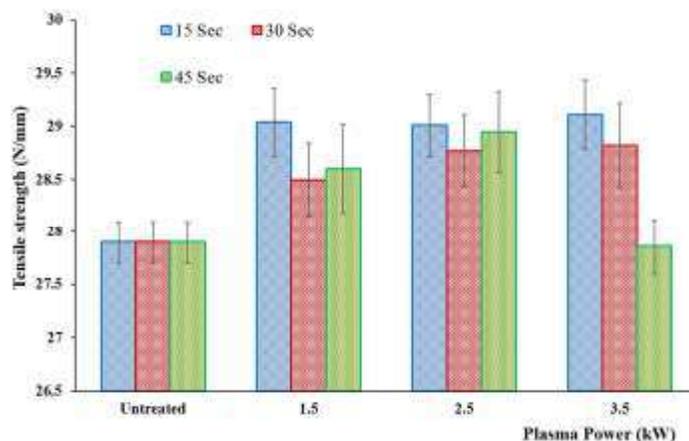


Figure 4: Tensile strength of plasma-treated nylon fabric

interaction may cause an increase in tensile strength by increasing cohesive force between the fibres [13].

Conclusions:

Plasma treatment of nylon 66 fabric was carried out in atmospheric pressure plasma using helium gas. It was found that after plasma treatment the wettability was improved and adhesion with the coating is also improved. Further, it was noticed that the change in wettability and adhesion strength are highly dependent on plasma processing parameters. The plasma exposure time of 30sec and 2.5kW plasma power was found to optimum for maximum improvement in adhesion strength. No adverse effect on tensile properties was seen after plasma treatment.

Acknowledgement

The author is thankful to the Ministry of Textiles, Government of India, for providing financial assistance for this study.

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Scope and Opportunities of Textile and Garment Development in Odisha

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Abstract

The Handloom, power loom, and Garment sectors are becoming vital industries day by day because of the employment generation, socio-economic problems, and revenue generation from the Export market. Odisha being a rice-producing state with mines and heavy Engineering Industries, not much importance was given to the Textile Sectors. There are neither organized sectors nor any Composite Mills, not even a single Spg Mill is working because of the raw material price (cotton) and lack in technological aspects. The small scales sectors are facing problems of the high price of yarns with high transportation costs, unskilled workforce, apathy to work hard, less ambitious people, nor any accurate supports from the Central and State Govts. This paper has discussed the pros and cons of each sector with solutions.

Key Words:

Handloom, Power Looms, Garments, Skill, marketing

1. Introduction:

Odisha is an agriculture-based economy that is in the transition towards industry and service-based economy. According to recent estimates, the size of Odisha's economy has increased by 122.27% during the last six years in terms of the gross state domestic product (GSDP). Thereby, Odisha achieved an annual average growth rate of 6.23% during that period. Odisha had a rural unemployment rate of 8.7% and an urban unemployment rate of 5.8% calculated based on the current daily status basis in the 68th National Sample Survey (2011-2012)¹. As per the figure in November'20, the unemployment rate in Odisha is 1.7%⁽²⁾.

According to the 2011 census of India, 61.8% of the working population is engaged in agricultural activities. However, the agricultural activities undertaken in the state contributed only 16.3% to the GSDP in the fiscal year 2013-14 and it was estimated to be 15.4% in 2014-15. Rice is the dominant crop in Odisha. It is grown on 77% of the area under cultivation. According to the 2011 census of India, 61.8% of the working population is engaged in agricultural activities. However, the agricultural activities undertaken in the state contributed only 16.3% to the GSDP in the fiscal year 2013-14 and it was estimated to be 15.4% in 2014-15. Rice is the dominant crop in Odisha. It is grown on 77% of the area under cultivation.

The primary industries in Odisha are manufacturing; mining and quarrying; electricity, gas and water supply, and construction. The industrial sector's contribution to the state's GSDP was estimated at 33.45% in 2014-15. Most of Odisha's industries are mineral-based. Odisha has 25% of India's iron reserves. It has 10% of India's production capacity in steel. Odisha is the top aluminum-producing state in India. Two of the largest aluminum plants in India are in Odisha, NALCO, and Vedanta Resources. Mining contributed an estimated 6.31% to the GSDP.³ Odisha has 9036.36 MW installed capacity of electricity production, out of which 6753.04 MW is coal-generated. 2166.93 MW is generated by hydro-power and 116.39 MW by other renewable sources.

The service sector contributed an estimated 51% to the GSDP in 2014-15. The primary sub-sectors are community, social and personal services, which contributed 13.45% to the GSDP; trade, hotels, and restaurants, which contributed 13.09%; financial and insurance services, which contributed 13.64%; and transport, storage, and communication, which contributed 10.99%. The state has a well-developed banking network compared to many states of India. There is one bank branch for every 12,000 people. 90% of the branches are in the rural region.¹

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2. Textile at present

Handlooms, Textiles, and garment Industries have a very gloomy picture in the State and it requires development. The present state can be summarized as follow:

1. State produces only 3% of the required clothes. No power loom units or spinning mills have been established in Odisha in the last 10 years. No investment proposals yet.
2. Biju Babu's Dream Project OTM (Odisha Textiles Mill), 12 other power loom units, and 13 spinning mills have been shut down.
3. Another constraint found of the unskilled and lazy workforce (market survey).
4. Unfortunately, the Handloom Industries are surviving only from the Support from the Governments.
5. Odisha produces high-quality cotton.
6. Odisha needs 108 crore sq. ft. clothes @ Rs 12,000 crore every year⁽⁴⁾.

To develop rural areas, for more employment generation among the low qualified people in Odisha, it is necessary to develop the Textile Sectors (Handloom, Power loom and Garment) and it will earn revenue for Export Market too. It will generate more state GDP also. Because of the more employment generation in this sector, the people of Odisha will remain in the state rather than go to the other different states like Tirupur, Surat, Silvassa, Bhiwandi area to earn their bread & butter.

2.1 Handloom in Odisha: present scenario

Handloom Sector, next to agriculture provides massive employment to the rural artisans. Handloom cloth is one of the richest and resilient mediums of ethnic expressions. For the weaver weaving of cloth is not just a commercial venture but it represents the philosophy of the way of life. It shows the Art, the Culture, the history of that region, and that's why Bangladesh is coming forward to reproduce Jamdani or Dhakai Muslin.

The Handloom Industry in Orissa is the largest cottage industry providing employment and sustenance to 4 % of the population of the State. However, in face of teething competition in the open market, the strengthening of the Industry and its diversification is the continuous need for its survival. Today it is having a 1.19 lakh number of looms in the state⁽⁵⁾. Of which 88186 nos of looms have been brought under the cooperative fold and developmental activities are mostly being undertaken in this organized sector under the directorate of Textiles.

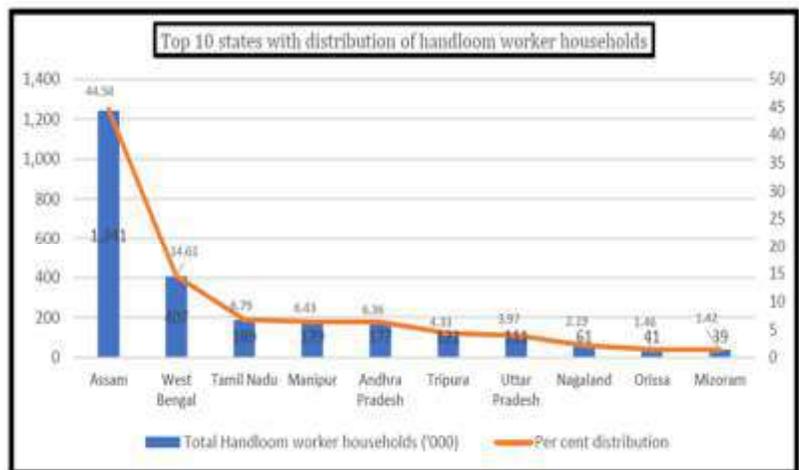
Figure 1 shows the famous Handloom sarees produced in the State. The Brand names are; Berhampuri Saree, Khandua

Saree, Pasapali Saree, Bomkai or Sonepuri Saree, Sambalpuri, Ikat, or Bandha Saree. They are unique but how much common people can afford it? It is known as Elite class Sarees.



Figure 1: Odisha Handloom Sarees

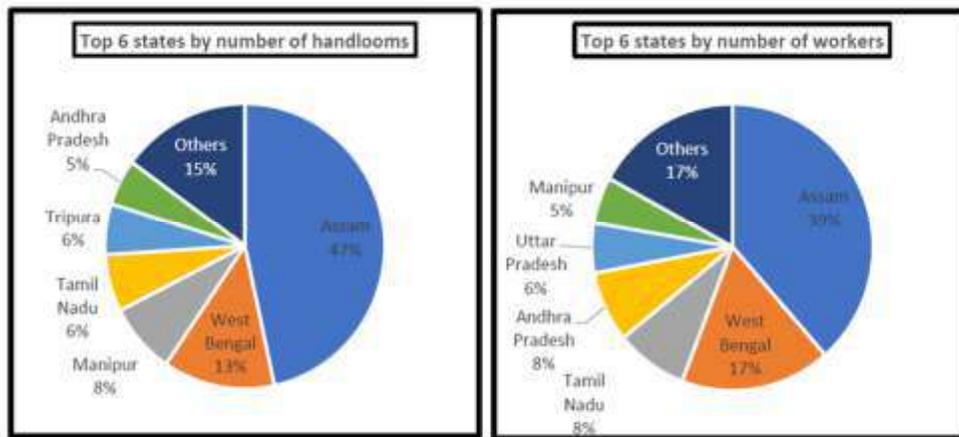
The Saree is becoming out of the fashion nowadays. The new generation prefers to wear jeans, dress material which are comfortable in day-to-day life, because of cheaper prices, easy maintenance, washing and ironing, easy handling while going to schools, colleges, and working places. Such sarees are being costly are worn during festivals and ceremonies as Fashion and to show Aristocracy. These high-quality sarees are not only confined in Odisha but the major buyers are from Bengal, North East, and the Northern States and in Bangladesh too. But unfortunately, the Odisha Brand Name "Boyanika" is not found in major cities including Mumbai, Nagpur, Pune, Ahmedabad, and Jaipur, and even in Bangladesh where the market potential is very good. (Market research).



Source: TARI Research Team, based on Third Handlooms Census, Government of India

Figure 2: Handloom Production in India

Figure 2 shows that only 1.46% of the people are engaged as Handloom Workers in Odisha. Figure 3 shows the number of Handlooms and Handloom weavers is too less in comparison to other states like Assam, West Bengal, and TN. The causes are obvious that this industry produces the majority of



Source: TARI Research Team, based on Third Handloom Census, Government of India

Figure 3: Handloom Sector in India

traditional clothes like Gamcha, blouse pieces, Dhooty, lungi but because of cut-throat competition of the similar products from the other states, they are suffering. There is no standard quality, nor any checking nor any inspection system even for the high standard quality sarees and hence the rejection is higher. There are rising hopes that today apart from “Boyonica”, there are “SambalpuriBastralaya”, “Utkalika” who are exporting Online, and the Handloom industries in Odisha are slowly progressing and gradually facing challenges.

2.2. Handicaps of handloom today:

Odisha handloom faces various problems at present and it can be summarized as follows:

2.2.1. Raw material availability:

The contribution of Odisha towards India's total cotton production is barely 0.5%, the cotton produced in the state is of exportable quality⁽⁶⁾. Odisha Produced 400 thousand tons against 9,700 thousand tons of Gujarat⁽⁷⁾. Hence it is necessary to transport cotton from Gujarat and Maharashtra that fetches a high price. Hence the cost of the products becomes higher.

2.2.2. The usage of Dyes & Chemicals:

The Handloom weavers are not very thorough in using reactive dyes, vat dyes, and Acid Dyes to be used in Silk. They are not aware of the process parameters, hence there is huge wastage of dyes & Chemicals, improper dye fixation, increasing expenditures. At Some places, they use Branded Dyestuff like Colourtex but in most cases, it increases cost without achieving proper results. Like TN, low-temperature low-pressure dyes can be used. (Market research). There is no professional Process house for dyeing systemically at a low cost. They can be encouraged to use Natural Dyes also.

However, at some places like Nuapatana, Athgarh, Mania Bandha the Clusters are formed group-wise where the Handloom weavers get services like arrangement of Raw materials, Dyeing facilities from ADT (Asst Director, Textile), who controls the Clusters. But more updated technology is required for the developments and market competitions. (Market survey).

2.2.3. Skill

Skill is another important factor and despite having sufficient Manpower, Odisha faces a shortage of skilled manpower. NHDC and the officers are not interfering in such matters (market survey). There are Experts available to improve the skill of the Handloom weavers especially senior Industrial workers, senior artisans who can also develop new and newer designs but it all must be initiated by the top-level Govt officials. The Training Centre is more expensive than the positive outcome (Market survey)

2.2.4. Use of Polyester Yarn:

It is in use but in low profile. There is not much selling of Polyester yarn in the Odisha market. It is hardly sold 5-7 MT/month mostly in Handloom Sarees and a little in Lungi, Blouse pc, etc. The weavers are neither much aware of the know-how of the usage of polyester yarn nor aware of the end-user. (Market survey). At some places “Polyester bastra” are manufactured (warp polyester, weft Cotton) for Saree and shirting but is not very popular as per the market conception that Handloom means the only Cotton. (Market survey).

In Dhenkanal, the staple fibre production unit by RIL was stopped in the year 2012 (production capacity was about 100 MT/day) because of poor marketing opportunities in Odisha (Market Survey) and the high cost of production. The future of Polyester yarn (staple and continuous Filaments) will be easier in Odisha after the manufacturing of the same from Paradip and Bhadrak (IOC Project).

2.2.5. Lack of update Consumer Preferences Products:

The Sambalpuri ikat and others have a manageable market but the other handloom production like a napkin, Lungi, towels, etc. have failed to draw the attention of the people of local and outside. The weavers do not have the opportunity to upgrade those products based on the demand of the people. As a result, the weavers have been continuing to produce traditional productions without having the novelty of designs, colour combinations, and texture. No special training program has been organized by the government or

cooperatives societies to upgrade the skill-based on consumer choice, though the handloom policy 2016 emphasis promoting capacity building through skill up-gradation and institutional training (Handloom Policy-2016).

2.2.6. No Textile Parks:

As per the Press Information Bureau, Govt of India, Ministry of Textile, there is a total of 61 Textile parks in India (2012-17, more are yet to come), there are 15 parks in Maharashtra, 9 in Rajasthan, 7 in AP, 10 in TN, 8 in Gujarat, 1 each at MP, Tripura, HP, J&K, 3 in Punjab, 2 each in W.B. & Karnataka but Nothing is in Odisha. As per the new financial year budget 21-22, one is expected to come at Bhardak(Market research).

2.2.7. Lack of Marketing Facilities:

Apart from online export facilities developed and exhibitions arranged there should be more showrooms of “Bayonika” “ Utkalika” “SambalpuriBastralaya” with dynamic Salesmanship at more places in cosmopolitan cities with publicity. The Barpali cluster has a good weekly market facility called “Behera Market” locally known as “Baeljuri Bazaar”, situated 8 km from Barpali town. Only local handloom products are available there. The market is open in the early morning i.e., 4.00 am to till late evening of Friday. As the marketplace is situated at the side of the national highway No. 201, it connects all the district headquarters of West Odisha and capital cities Bhubaneswar and Raipur directly through the road. The railway station of Barpali has situated approximately 8km from the Baeljuri bazaar. To marketing support, the said bazaar should be strengthened through advertisement in printing, electronic media, and add with tourism website. Moreover, marketing tie-up may undertake with other marketing agencies or export agencies. The deadly cooperative societies may be financially assisted to grow sales and enhance daily earnings. To make an online purchase system the necessary facilities such as printing of product catalog, sale outlets, and website for easy accessing may be arranged.

2.3. Handicaps of the powerloom today

Unlike Bhiwandi, Surat, Ichalkaranji, the condition of the Powerloom shows a very gloomy picture. It is hardly 2000 Ordinary looms (over pick & under pick) (market survey)

There are private powerloom units spread over different parts of Odisha like Siminoi, Itamati, Hinjilikatu, Takatpur, Nayagarh, etc. It is observed that all the small entrepreneur are running their units with a very small number of looms in their Home shed. They are only depending on the local market⁽⁸⁾. Most of them consider it as their domestic business. Some looms are as old as 50 years. No maintenance, no quality consciousness, and no new innovativeness no technical know-how are there. They prepare beams of their homes in their way on the wooden drums maintaining no quality.

Most of them are producing Gamucha and blouse pieces with 20's warp, 20/26/30's cotton weft, and 40 reed / 40 picks per

inch. (Market survey). Some Entrepreneur are having 20 looms or more and willing to put China Rapier looms and willing to produce Shirting, Voiles, etc but because of poor marketing opportunities, they are not coming forward. (Market survey)

It is high time to prepare beams with modern technology located at any central place/ service center from where the beams can be reached at Weaver's home with lesser transport cost. Let them be guided to produce value-added products with proper guidance of selling procedures. There should be Dobby/Jacquard on the power looms to produce value-added Gamucha as shown in Picture 4 below. This type of Product with verities of Jacquard Design will be highly valued and appreciated throughout India. The Private entrepreneurs and/or upcoming entrepreneurs can take this as an initiative to sale. Power loom service center should also come forward in support of value-added production.



Figure 4: Design Gamuchha

Garment sectors in Odisha

The garment is one of the identified focus sectors of Odisha as a part of their Vision 2025 for Industrial Development. In addition to the incentives extended by the Industrial Policy Resolution 2015 of the State which identified the Textile and apparel sector as the priority sector, the State has promulgated Odisha Apparel Policy 2016 to provide support for the development of the sector.

Recent/Planned Investments in Apparel Sector:

Shahi Exports Pvt. Limited, the largest manufacturer and exporter of garments in India, started its operation in December 2016 in Mancheswar Industrial Estate, Bhubaneswar. The unit currently employs about 2,000 people of which 95% are women. The unit has the capacity of producing approx. 4 million pieces per month. They are also planning to establish a second factory nearby. They also have plans to start two shifts here and have been in talks with Labour Ministry for the same. Shahi has plans to make the factory in Bhubaneswar the best apparel factory in India. Aditya Birla group has also started construction work for setting up of apparel manufacturing unit in Bhubaneswar with a capacity of 2.5 million pieces per annum. It is estimated to generate employment for about 1,600 people. The government has identified the following locations for apparel manufacturing in Odisha: Ramdasapur, Cuttack (173

acres land) - Page Industries (Jockey) has plans to set up a manufacturing facility there. . (Posted by [Editorial Team](#) | Aug 22, 2019, | [Domestic](#)). Chhatabar Apparel Park, Khurda (100 acres), Jayamangal, Khorda (129 acres) Tata Industrial Park, Gopalpur (3,000 acres) Damnagar, Bhadak (100 acres)⁽⁹⁾. (most of them are under proposed)

A report of the Apparel Export Promotion Council (AEPCC) titled 'Location Analysis for Selected Cities in Odisha for Apparel Sector' shows that Odisha is emerging as one of the top destinations for apparel manufacturing in the country. The State Government has identified apparel as one of the identified focus sectors as part of its Vision 2025 for industrial development.

Almost all the Garment units have common problems that they are associated with a shortage of raw materials (threads, buttons, proper fabrics), problems of marketing, non-availability of working capital, competition from large scale industries, problems of labour, lack of up-gradation in Technology, poor support from Govt. etc.

Conclusions

1. Odisha is the State of Agriculture, Mines, heavy engineering, and Ports and hence the Unemployment is very less.

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2. But to generate employment among the low qualified people and for the socio-economic problems, it is necessary to grow Handloom, Power loom and Garment Sectors. It will fetch Export and local market too.
3. The Handloom sector is producing highly fascinated and world-famous fabrics but the users are the elite class of the society who has got more options and choices too.
4. The traditional cloths i.e Gamucha, Lungi, blouse pcs are produced but the sales & marketing are not adequate. More Technical supports are needed to modernise the same.
5. The power loom industries, though coming up but it is a homemade Industry. Entrepreneurship is to be encouraged. Auto looms, good warping machines are to be installed to increase the lively hood of the weavers.
6. The people are also less ambitious hence do not welcome any new Technology.
7. The Garment Sectors are also growing with Private Entrepreneurships but because of the constrains like shortage of raw materials and skilled workforce, it is getting struck off.

BTRA Facility :

Thermogravimetric Analysis (TGA) :

Thermogravimetric Analysis (TGA) is a method of thermal analysis in which the weight loss or gain is measured as a function of temperature or time.

This measurement provides information about physical phenomena such as phase transition, as well as chemical phenomena including thermal decomposition.

Some of the important application of TGA is to find:

- 1) Moisture and volatile content,
- 2) Composition of polymer, Filler and carbon content.
- 3) Thermal stability of Material
- 4) Decomposition kinetics of material

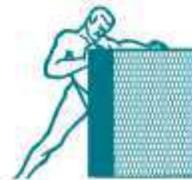
We have SDTQ600 model in which we can capture simultaneously, DSC as well as TGA signal. In normal DSC we can go only upto 500°C, but the advantage of this instrument is that we can get the DSC signal upto 1200°C



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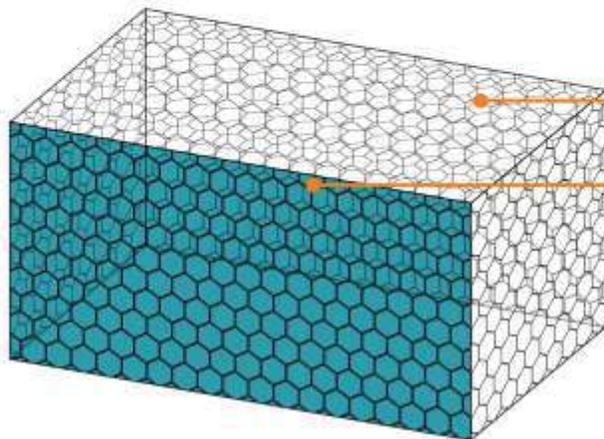


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