CAUSAL ANALYSIS OF PROCESS VARIABILITY IN DOPE PREPARATION SECTION OF LYOCELL FIBER MANUFACTURING PROCESS AND RECOMMENDATIONS FOR IMPROVEMENT

A MAJOR PROJECT REPORT

Submitted in partial fulfillment of the requirements for the award of the degree

of

Master of Technology

in

CHEMICAL ENGINEERING

(With Specialization in Process Design Engineering)

By

DIBYARUP MAJUMDAR

DEPARTMENT OF CHEMICAL ENGINEERING

UNIVERSITY OF PETROLEUM & ENERGY STUDIES, DEHRADUN

UTTARAKHAND-248007, INDIA

April -2016

CANDIDATE'S DECLARATION

I hereby declare that the work, which is being presented in this dissertation, entitled **"Causal analysis of process variability in dope preparation section of Lyocell fiber manufacturing process and recommendations for improvement'',** submitted in partial fulfillment of the requirement for award of the degree of **Master of Technology** in **Chemical Engineering** with the specialization in **Process Design Engineering (PDE),** is an authentic record of my own work carried out under the supervision of **Dr. Ashutosh Pandey**, Professor, Department of Chemical Engineering, University of Petroleum & Energy Studies, Dehradun and **Dr. Parag Patil,** Assistant Vice-president, PFIC, Grasim Industries Limited, Taloja, Maharashtra from industry.

Date - April, 2016 **Dibyarup Majumdar**

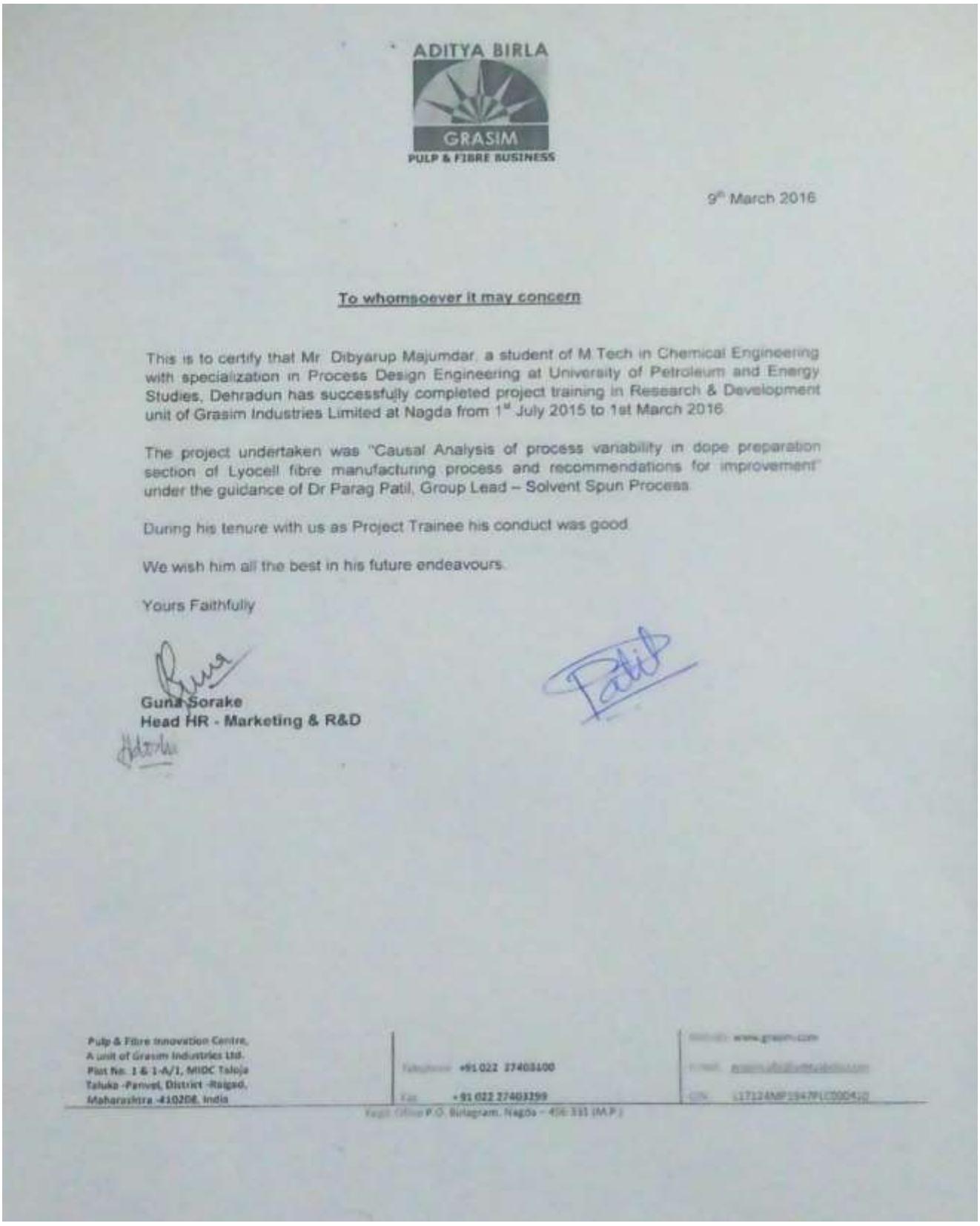
Place – Dehradun **Reg: R670214023, SAP ID: 500033012**

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

Dr. Ashutosh Pandey
Professor,
Department of Chemical Engineering
University of Petroleum & Energy Studies
Dehradun, Uttarakhand, India-248007

Head of the Department

INTERNSHIP COMPLETION CERTIFICATE



ADITYA BIRLA
GRASIM
PULP & FIBRE BUSINESS

9th March 2016

To whomsoever it may concern

This is to certify that Mr Dibyarup Majumdar, a student of M Tech in Chemical Engineering with specialization in Process Design Engineering at University of Petroleum and Energy Studies, Dehradun has successfully completed project training in Research & Development unit of Grasim Industries Limited at Nagda from 1st July 2015 to 1st March 2016.

The project undertaken was "Causal Analysis of process variability in dope preparation section of Lyocell fibre manufacturing process and recommendations for improvement" under the guidance of Dr Parag Patil, Group Lead – Solvent Spun Process.

During his tenure with us as Project Trainee his conduct was good.

We wish him all the best in his future endeavours.

Yours Faithfully

Guna Sorake
Head HR - Marketing & R&D

Pulp & Fibre Innovation Centre,
A unit of Grasim Industries Ltd.
Plot No. 1 & 1-A/1, MIDC Taloja
Taluka -Panvel, District -Raigad,
Maharashtra -410208, India

+91 022 27403100
+91 022 27403299

www.grasim.com

Regd. Office P.O. Birlagram, Nagda - 456 331 (M.P.)

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Dated: April , 2016 **Dibyarup Majumdar**

Place: Dehradun

NOMENCLATURE/ABBREVIATIONS

0.5/10 TPD- 0.5/10 Tons per day

Cp- Centipoise

WFE- Wiped Film Evaporator

Dope- Reddish mass obtained at the outlet of LIST/Wiped Film Evaporator

RI- Refractive Index

MFI- Melt Flow Index

DP- Degree of polymerization

SP- Set point

PV- Process Value

CTQ- Critical to Quality

ABSTRACT

The primary objective of the project titled 'Causal analysis of process variability in dope preparation section of Lyocell fiber manufacturing process and recommendations for improvement' was to both quantitatively and qualitatively reduce the perturbations of the critical parameters of the process. The scope of the project revolved around the pulper and the Wiped Film Evaporator (WFE) / LIST section. The work done till date consists of two production division viz. 0.5 TPD and 10 TPD. The major parameter considered for study was Melt Flow Index (MFI) as this is one of the Critical to Quality parameters of dope. The factors directly/indirectly affecting MFI were studied and correlations were deduced. A sampling exercise was also conducted to determine certain properties of pulp, slurry and dope of 10 TPD section viz. Cp, Degree of Polymerization (DP), % Cellulose in slurry, dope and MFI of dope experimentally. The results obtained were analyzed and discussed in detail. Certain problems were observed in both 0.5 TPD and 10 TPD and recommendations to mitigate those have been proposed.

Keywords: *perturbations, Melt Flow Index, Wiped Film Evaporator, cellulose, NMMO*

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1. OVERALL OBJECTIVE OF THE PROJECT

The most critical customer requirement in lyocell fiber is to maintain consistency in fiber quality and properties. This requires maintaining a consistent dope quality over time with minimum variation in dope composition and rheology. All critical process parameters in the dope preparation step should be maintained in a narrow range to achieve this. The plant has identified the critical parameters in dope preparation section and also the range in which these parameters should be maintained. These parameters are being tracked on a continuous basis through Historian system. Analysis of past data indicates significant process variability with many of these critical parameters being out of range for substantial time. The objectives of this work is to understand the root causes for disturbances resulting in high process variability and identify solutions to address the same. The work broadly consists of following steps

- Define characteristics of 'good' dope required for stable spinning and desired fiber quality
- Understand cause and effect relationship between process parameters and dope quality through analysis of process data
- Root cause analysis to identify the main reasons for disturbances in the process of dope preparation and evaluating whether it is a/an -
  a. Design deficiency
  b. Inappropriate process parameter setting
  c. Inadequate process control
  d. Inadequate definition and follow-up of Standard Operating Procedures (SOP's)
  e. Human Error
- Identify and recommend solutions to the problems deduced

2. INTRODUCTION

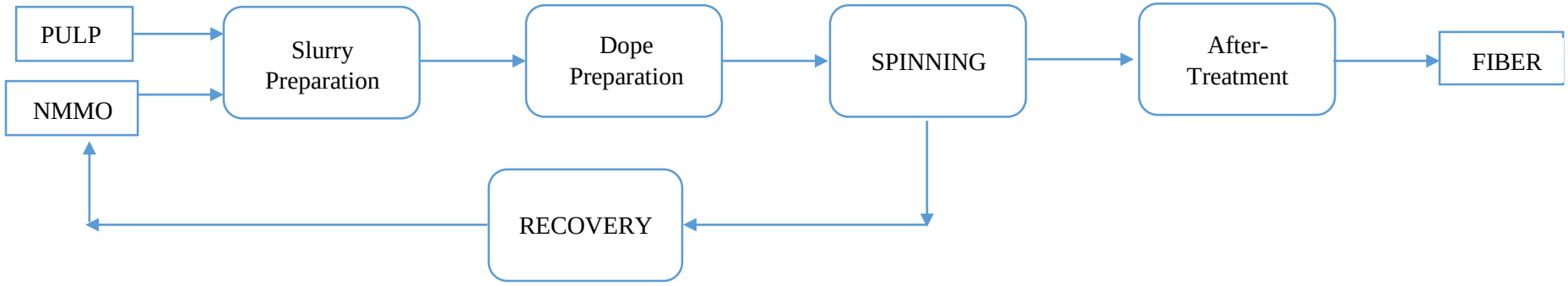
2.1 Process History:

Cellulose is an organic compound with the formula $(C_6H_{10}O_5)_n$, a polysaccharide consisting of a linear chain of several hundred to many thousands of glucose units. [1] Cellulose is one of the most abundant natural resources on earth and has been used as a raw material for several decades. Cellulose fiber history dates back to the 1860s, when the first rayon fibers were commercialized by Courtaulds. [2] The lyocell fiber has a highly crystalline structure in which crystalline domains are continuously dispersed along the fiber axis. This offers good wet strength as well as excellent dry strength, which makes lyocell water-washable. Further, it shrinks less when wetted by water and drier than other cellulose fibers such as cotton and Viscose. [3, 4, 5, 6] Lyocell process uses NMMO hydrates with a hydration number (n) greater than 1.[7, 8] It also affects the physical properties of the fibers spun from the solution.[9,10] NMMO has emerged as the best of amine-oxides and Neil Franks and Julianna Varga[11] developed a way of making a more concentrated and hence economical solution of cellulose. The dissolving power of tertiary amine oxides for cellulose achieved greater interest only during the late sixties, when the dissolution of cellulose in tertiary amine Oxides, N-methylmorpholine-N-oxide being among them, was detected. [12]

Paper named '*Investigations for preparation of cellulose solutions in NMMO and the following forming*' by Dr. Reinhard Maron, Dr. Cristhof Michels, and Dr. Eberhard Taeger from Thuringisches Institute for Textile, Germany published in Lezinger Berichte, 9/94 had one of the biggest contribution towards development of the project and commissioning of this plant.

2.2 Brief Process description:

This process basically has two raw materials viz. NMMO and Cellulose. Cellulose pulp is directly dissolved in NMMO solvent and made into a slurry in the pulper. This slurry is converted to polymer solution (dope) in a Wiped Film Evaporator/LIST. The dope formed is then sent to the spinning section after filtration. The fibers are then sent to the after- treatment section for washing, bleaching and surface finish application followed by drying section. The finished fibers are then packed into bales. The NMMO recovery section is very important here because > 99% of NMMO is recovered and recycled and therefore it is considered an environment friendly process.



2.3 Overall Block Flow diagram for the process:

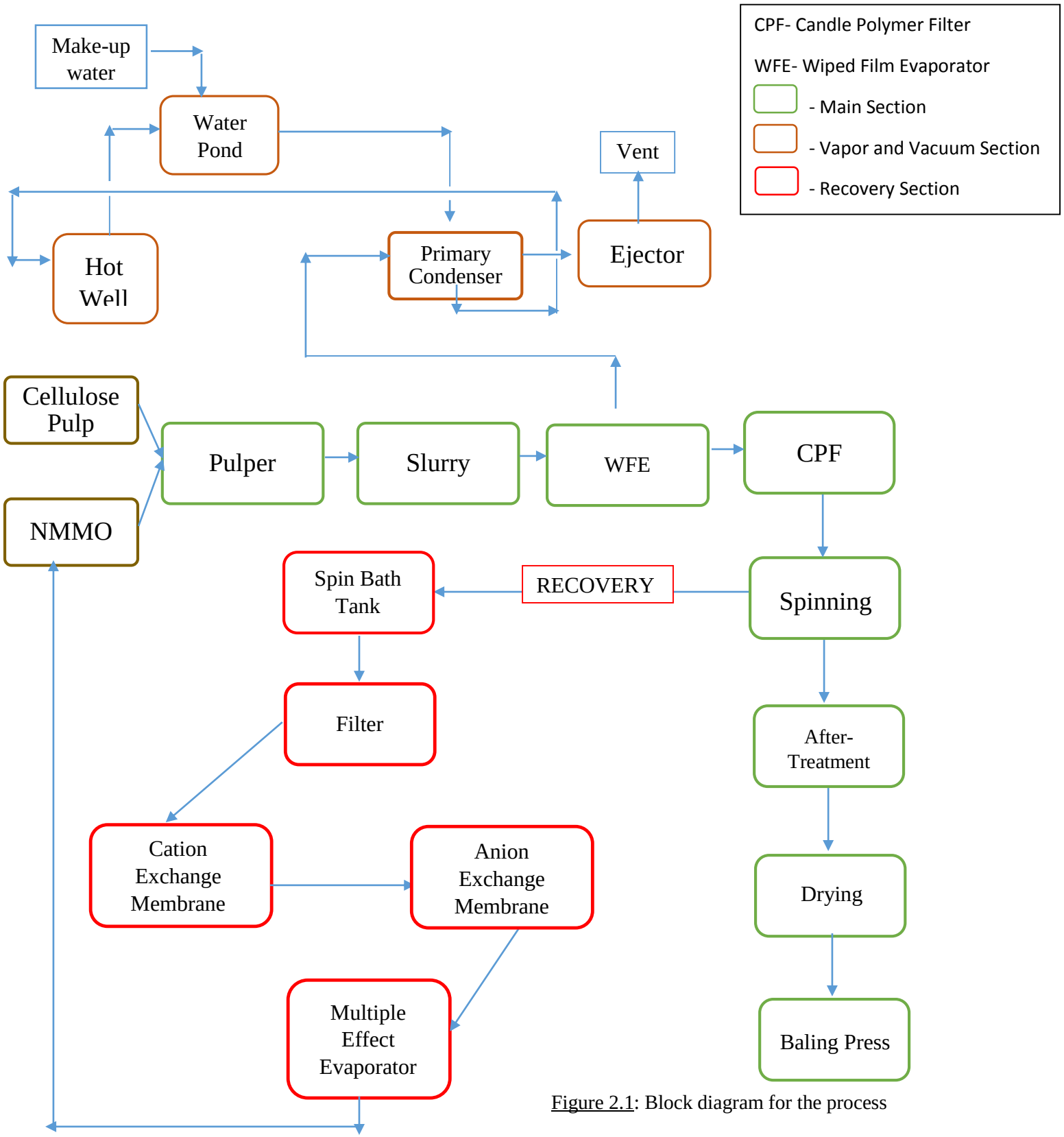


Figure 2.1: Block diagram for the process

3. STUDY OF 0.5 TPD PILOT PLANT

3.1 Process Description:

3.1.1 Pulper

The pulper in the 0.5 TPD is similar in operation to the pulper present in 10 TPD operation. As the production rate is less, there is only one pulper installed. Depending on the cellulose % desired in the dope coming out of the LIST, we decide on the pulp weight to be fed to the pulper. The NMMO used is around 240 L (276 kg) which is supplied from a tank near the pulper. This tank receives NMMO from the recovery section which runs NMMO in circulation. The NMMO line is jacketed by LP steam and generally supplies NMMO at 62-95 ^{0}C. About 80-100 g of PG (Propyl Gallate) is added per batch. There is a timer installed in the pulper which ensures that each batch is run for 40 minutes. Contrary to the pulper in 10 TPD, the pulper here in 0.5 TPD is externally jacketed. The heating circuit present pumps hot water to the hopper followed by the pulper and then run in the jacket of the NMMO vessel and recirculated. The instrumentation is controlled from the DCS panel installed near the spinneret. The batch formed is immediately dumped in the hopper. The batch is usually made when the hopper has the level (storage) of 1.5 batch. There is a RTD installed at the base of the hopper. The RTD has its probe inserted in the slurry.

3.1.2 LIST

The slurry enters the LIST through a DKD system installed at the bottom of the hopper. This has a piston-cylinder arrangement which involves a suction and a discharge stroke. The suction stroke pulls in the slurry from the hopper while in the discharge stroke the slurry is pushed out to the LIST. There are two parameters associated with this. First is Cycle time and secondly Number of strokes. The cycle time primarily decides the amount of slurry sent to the LIST. If it is high, less amount of slurry will be sent and vice-versa. The value for number of strokes is recorded for every 24 hours after which it restarts from zero. Generally around 550-650 strokes takes place in a day. Now the slurry enters in the LIST. The LIST has two shells as a part of heating circuit where hot water is circulated to supply heat to the slurry for moisture evaporation. The LIST internally has four RTD's placed at four hypothetically decided zones. These zones are made at each 25% length of the LIST. For rotating the blades in the LIST, a shaft moved by a rotor is installed. The rotor revolves at around 42-45 rpm. For supplying heat internally, hot water is circulated

inside the shaft. The shaft is pipe inside a pipe arrangement. The inner pipe sends water in the outer pipe whose outer surface is exposed to the slurry. The water is circulated by a centrifugal pump. The 4th zone is called discharge zone. Then there is ADS which is a screw pump and sends the slurry to the booster pump. This line is also jacketed using hot water. The rotor rotates making a film inside the LIST. The slurry is moved forward by the action of the blades. If there is more rotor rpm then this mechanical energy will be converted to thermal energy. The vapor coming out of the LIST is sent to an overhead condenser which is a shell and tube heat exchanger. The vapors are condensed by chilled water. The condensed vapors are collected in a condensate pot. The non-condensable gases are then sent to the steam ejector which gives vacuum to LIST. The remaining vapor are vented out. Several PHE's are installed for supplying hot water to-

- Hopper
- Shell I
- Shell II
- Discharge section
- Shaft
- Polymer line

These are used for maintaining the flow properties of the dope.

The evaporation zone temperatures are dependent on the Boiling point rise. According to experiments performed by the plant personnel, for 76.6 % NMMO concentration, the boiling point at 50 mm Hg is 77 ^{0}C. A calculation is exemplified below-

NMMO= (Weight of NMMO added * % concentration of NMMO) + (Pulp weight added * % Concentration of NMMO in pulp)

= (276 x 0.65 + 31.5 x 0) = 179.4 kg.

Moisture= (Weight of NMMO * % concentration of water in NMMO) + (Pulp weight added * % Concentration of moisture in pulp) = (276 x 0.35 + 31.5 x 0.08) = 99.12 kg.

So initial % of NMMO = (179.4/ (179.4 + 99.12)) x 100 = 64.411 % NMMO

Our target is to achieve 76 % of NMMO in polymer i.e. we have to concentrate the slurry and that is done by evaporation.

From steam tables we have,

760 mm Hg = 1 bar

50 mm Hg = 50/760 = 0.0657 bar

Now, on interpolation we have for 0.0657 bar the corresponding temperature from steam tables is 37.7 ^{0}C for pure water.

So, the Boiling point elevation is calculated from the data and the temperature to be supplied is 77-78 ^{0}C minimum for Zone 1.

3.1.3 Spinning/After-Treatment

The dope formed inside the LIST is sent to the CPF (Candle Polymer Filter) through the filter booster pump. Above the spinneret, there is the spinning pump which regulates the dope flow. There is hot water circulation in the spinneret to maintain the flow properties of the dope. There is the central line supplying air coming in from the AHU (Air Handling Unit) to facilitate the angular moving out of the fiber strands. The spinneret is installed over an NMMO dilute solution open tank like arrangement. This NMMO solution is maintained at a concentration of 24-25 % using the dope which contains NMMO flowing in from the modules. There is an air gap of 14-16 mm between the spinneret and the solution level. The spinning occurs in two stages- Dry spinning, Wet spinning. The spinning which takes place above the solution level is called Dry spinning while the spinning taking place below the solution level is called Wet spinning. Air quenching takes place in the process. The air gap maintained in the spinnerets facilitates the quenching process. When fiber strands come out, there is sudden cooling by the air passing. The regeneration of cellulose takes place in the wash. The fiber strands are then entangled together to form a tow and made to pass through different cylinder like arrangements via a guide where the fibers are towed to the cutter. While getting towed the fibers are rewashed with a dilute solution of NMMO (4-5% concentration). These processes are done in order to recover the maximum amount of NMMO. The fiber strands are then passed through a cutter feed roller into a cutter. A wash stream from cutter recirculation is run continuously to ensure moisture presence in the fiber strands before cutting. The cutter cuts the fiber strands into small segments. These are then sent to the after-treatment section.

The maintenance of concentration and level of dilute NMMO solution is quite interesting. The solution coming out from recovery trough and DM water wash is sent to the bottom

cutter circulation tank. Two lines from the cutter recirculation line are taken out. One is sent to the cutter to provide moistness and facilitate cutting operation. Second line goes to the tow wash tank where the spun fibers are washed. The overflow line from the tow wash is sent to the tow wash tank installed in the ground floor of the same building. The tow tank is connected at the middle to the 2nd spinning tank and the 2^{nd} spinning tank is connected to the 1^{st} spinning tank. The concentration in the spinning tank is kept around 20-24 %. This solution is sent to the open tank like arrangement below the spinneret at around 20 ^{0}C. This temperature is maintained by a PHE employing chilled water coming in from the chiller. A line from this line is connected with the recovery section where the NMMO is concentrated to about 64-65% employing distillation and sent to the pulper for batch preparation. The DM water wash flow rate should be kept same as the flow rate of NMMO solution sent to the recovery section.

The fibers after getting cut is sent to the recovery trough where LP steam is passed to open up the fiber. After the recovery trough the fiber are passed to the DM water wash to wash off the remaining NMMO. During this operation the fiber are sequentially passed through squeeze rollers to squeeze out the water. Now the fiber is passed through soft finish section to provide the desired softness, texture to the fiber. Then the fibers are passed through the Mat opener, sent to the dryer section and packed into bales.

3.2 Process Flow diagram with important instrumentation:

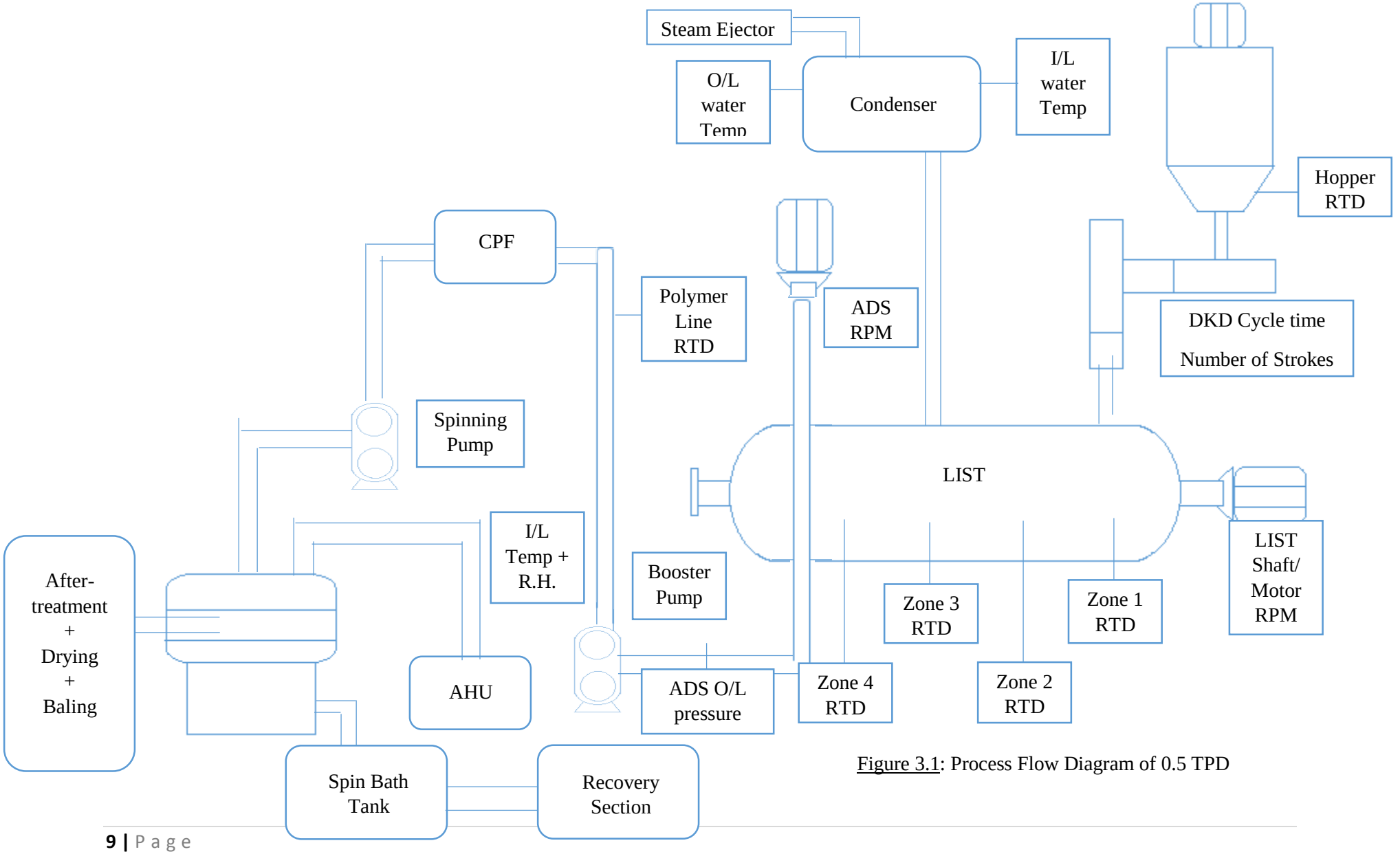


Figure 3.1: Process Flow Diagram of 0.5 TPD

3.3 Work done in 0.5 TPD pilot plant:

The primary parameters considered for study were-

- Hopper temperature (Slurry Temperature)
- DKD cycle time
- Hydraulic pressure for LIST rotor
- Vacuum maintained inside the LIST
- LIST Zone temperatures
- LIST rotor rpm
- Melt Flow Index (MFI)
- % Cellulose in dope

These parameters were decided keeping Melt Flow Index (MFI) as basis. The performance of the Dope preparation process is measured on the basis of MFI as this is one of the CTQ (Critical to quality) of the dope preparation. Subsequent data analysis have been conducted considering this as basis.

To understand the parameters the interdependence between them must be understood. So a few days were spent in understanding how the parameters were affecting each other and what control actions were being taken to keep them in the desired range.

Sample data were taken from the Historian system for analysis.

Time lag considered (For Data taken) - (6.08.15 8.00 pm – 24.08.15 4.00 am)

- Hopper Temperature= 2 hours prior.
- DKD Cycle Time= 2 hours prior.
- LIST Zone 1= 2 hours prior.
- LIST Zone 2= 2 hours prior.
- LIST Zone 3= 1 hour prior.
- LIST Zone 4= 1 hour prior.
- DTB Hydraulic Pressure = 1 hour prior.

- Polymer Temperature (Before Filter) = Current value.
- Candle Polymer Filter (CPF) I/L Pressure = Current value.

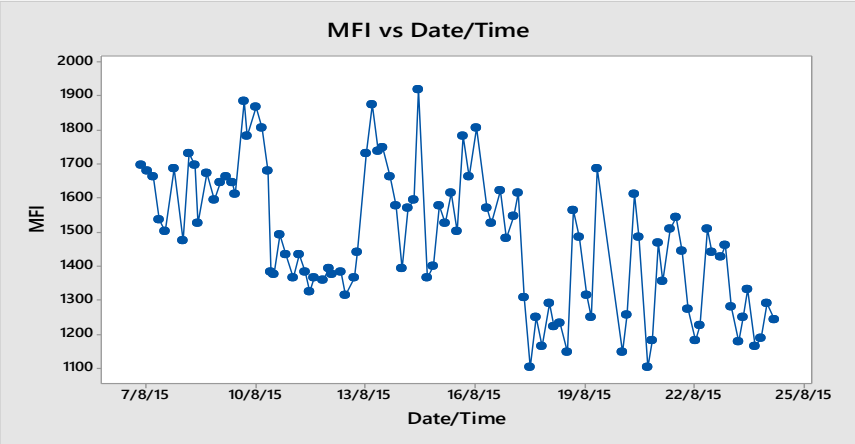
The following analysis have been conducted for the data over the period (6.08.15 8.00 pm – 24.08.15 4.00 am)-

a. Studying the variation of the process parameters against time
b. Correlation (Interdependence) study between the parameters
c. Regression analysis (STEPWISE) between the parameters
d. Normality analysis between the parameters
e. Variability analysis (Control chart) for individual parameter
f. Capability analysis for critical parameter
g. Plot displaying irregularity in Dope sampling interval
h. Studying the influence of Hopper temperature over other parameters

3.4 Results & Discussions:

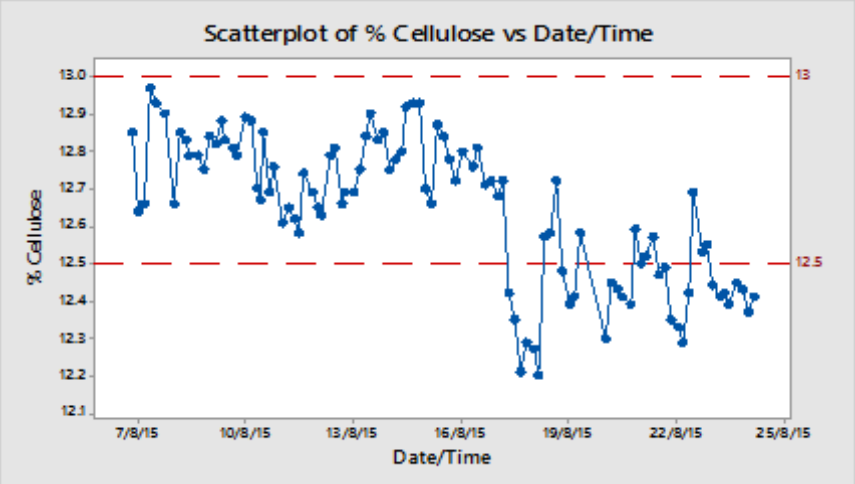
At the outset, Baseline data study pertaining to variation of individual parameters against time was conducted. The parameters selected were MFI, Hopper Temperature, LIST Zone 1 Temperature, LIST Zone 2 Temperature, LIST Zone 3 Temperature, LIST Zone 4 temperature. There are reference lines given for certain parameters which indicates the desired operating range.

a. MFI vs. Date/Time-

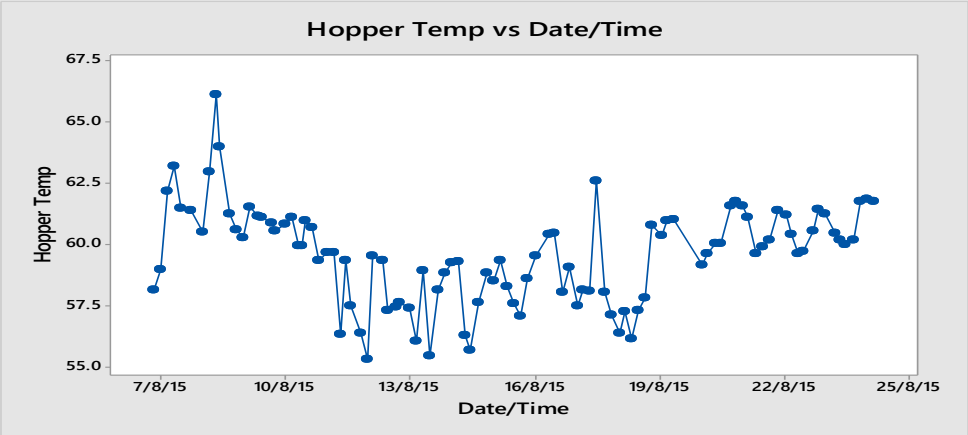


The MFI ranges in strict accordance with % Cellulose. So on changing % cellulose the MFI will change. During the period considered for the data, the % cellulose have been varied in between 12.5-13.0% but the MFI have been varying extensively.

b. % Cellulose-

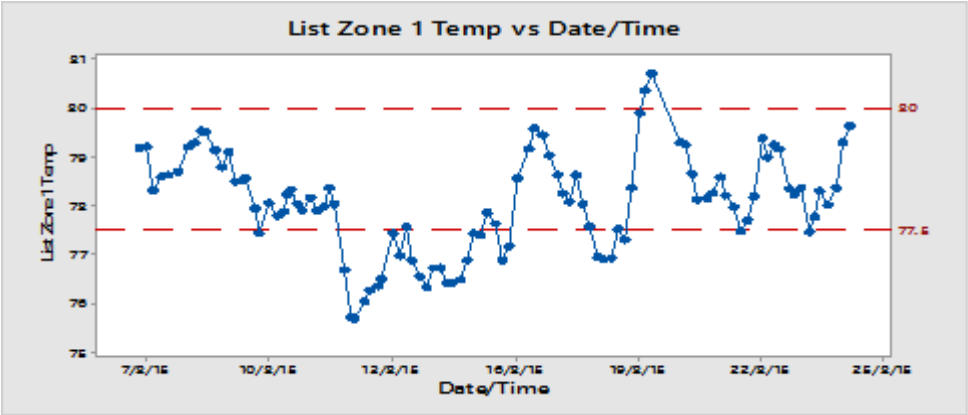


c. Hopper Temperature-



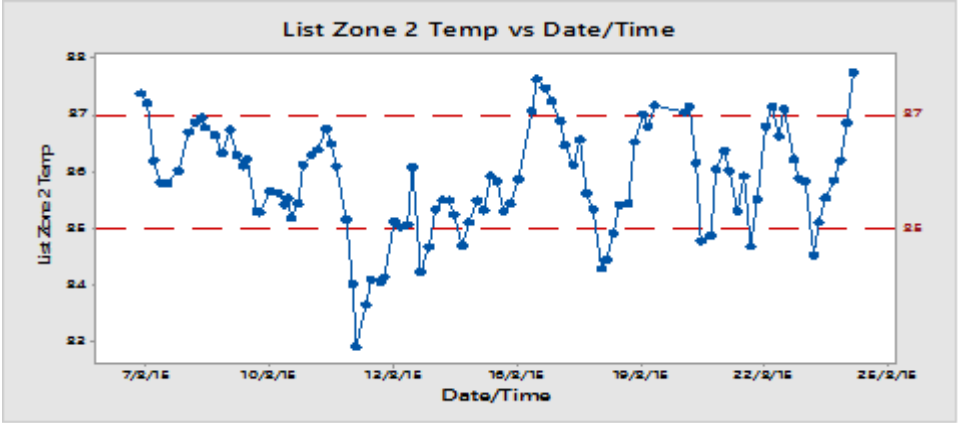
The hopper temperature has been thoroughly discussed in the subsequent pages.

d. LIST Zone 1 Temperature-

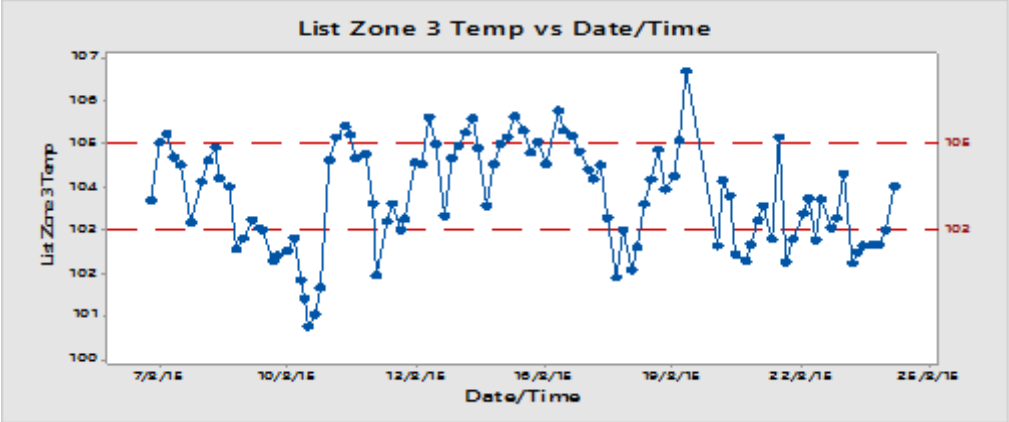


The above plot shows that there are major outliers on lower side. As per data analysis the variation in the Zone 1 temperature is because of variation in hopper temperature.

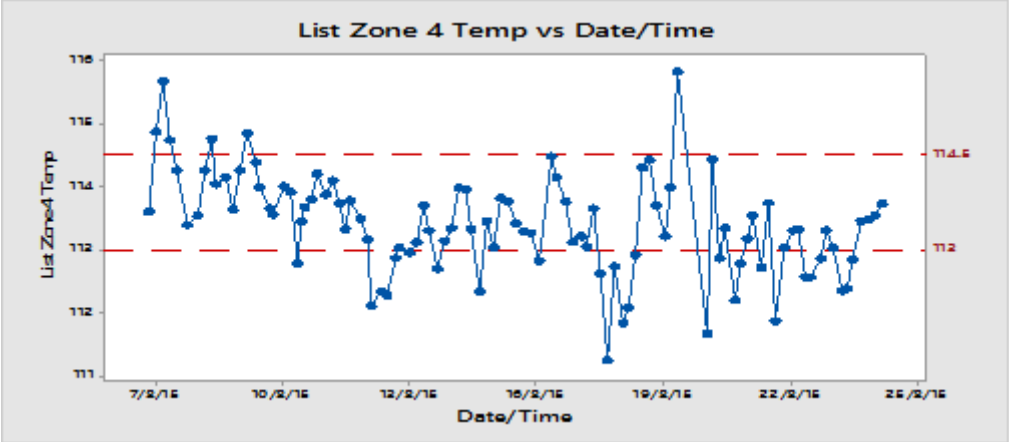
e. LIST Zone 2 Temperature-



f. LIST Zone 3 Temperature-



g. LIST Zone 4 Temperature-



Now the interdependence of the parameters are to be understood.

Interdependence of Parameters-

a) The hopper temperature is dependent on the slurry temperature. The RTD is installed at the bottom of the hopper. So when the slurry (85-95 ^{0}C) is dumped inside the hopper, it is hot at the top layers while the bottom layers from the previously dumped slurry is relatively cooler. So if the slurry temperature is hot, accordingly the hopper bottom temperature will vary.

b) The hydraulic pressure gives an idea about the holdup inside the LIST. If the hydraulic pressure increases, it indicates that there is increase in holdup inside. Then the cycle time

have to be increased i.e. more time will be taken for each cycle to complete which means less amount of slurry will be sent to the LIST. Even the temperature of the oil in the hydraulic system have to be maintained < 55 ^{0}C. If the temperature increases that means there is an increase in load over the hydraulic system which indirectly indicates that there is a holdup in the LIST.

c) The zone temperatures in the LIST depends on the vacuum maintained inside the LIST. This vacuum depends on the hopper level, cooling tower water supply.

d) The LIST rotor rpm is decided on the holdup in the LIST. But higher rpm will have a heat addition inside the LIST.

e) The shaft temperature is adjusted according to the temperature of the zones in the LIST.

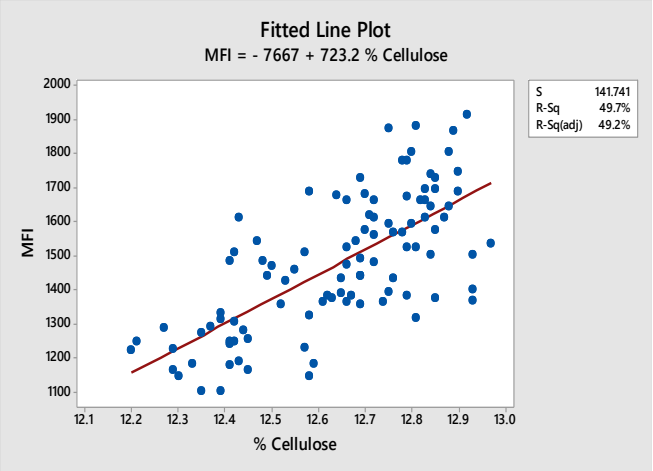
f) The discharge heating line temperature usually is adjusted according to the temperature of 4th zone.

g) The spinning line is also adjusted according to the temperature requirement of the dope flowing to the spinneret.

h) Generally the booster pump and the ADS discharge pressures are kept unchanged to ensure a constant flow of dope.

Fitted Line Plot for Parameters indicated in Correlation analysis-

% Cellulose vs. MFI-



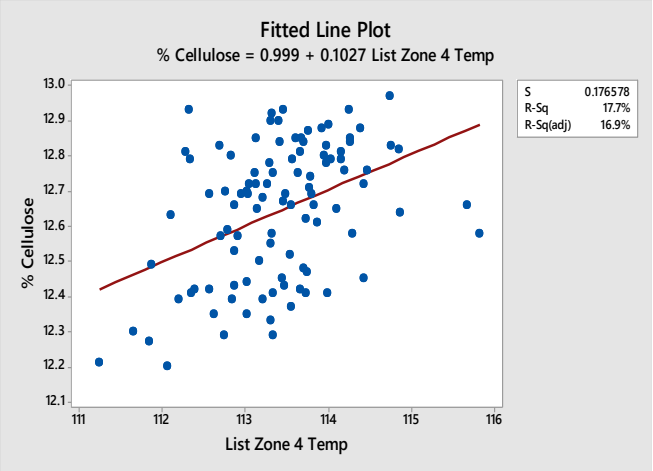


Fig: Fitted Line plot between MFI and % Cellulose, % Cellulose and LIST Zone 4 Temperature

Inference: With *reference to variation plots plotted against time*, all the mentioned parameters are observed to be varying. As per observation, the corrective actions taken should be applied sequentially and no parameter should be neglected. To maintain MFI, all of the processes are to be maintained properly starting from the slurry to dope preparation, then I believe that the process will be more or less constant in nature.

The following page consists of Table 3.1 where the observations and their proposed solutions have been recorded.

Gemba observation and proposed solutions-

Table 3.1: Observation and solutions of 0.5 TPD pilot plant

1. The NMMO line temperature which in the period (6.8.15 – 28.8.15) have been varying in the range 62-95 ^{0}C. The sampling study conducted previously clearly indicated that NMMO temperature has an effect on slurry temperature. Based on the influence of slurry temperature on other parameters (*refer study on hopper temperature below*), a clear effect is found on LIST Zone 1 temperature and DTB hydraulic pressure (*refer plot below*). Now in turn DTB hydraulic pressure has an effect on the vacuum, DKD cycle time etc. while Zone 1 affects the other Zones and these subsequently have an effect on the overall dope preparation process.	A PHE can be installed to ensure constant temperature of NMMO sent to the pulper for each batch preparation. If this is done then one parameter can be eliminated and we can move further down the process for other corrective actions.
2. Heating circuit temperature are varying frequently and these variations can generally be seen as a trend on the PC installed in the pilot plant. But the study of these variations could not be performed as the PC is non-functional for the past 2-3 weeks.	The heating circuit temperature has an impact on the dope preparation process as slurry to dope conversion is temperature reliant. Any study regarding the heating circuit temperature variation can only be followed after the PC is functional. Also the reason for this variation can be deduced and necessary corrective actions can be taken.
3. The hydraulic oil temperature should be maintained on the lower side (effect still not known)	The effect is not known but hydraulic oil temperature gives us an idea about the force exerted on the rotor and that in turn indicates the holdup in the LIST.

4. As per observation, the pulp consistency in terms of cellulose % and moisture content is questionable and should be looked into as it directly affects MFI (*refer plots above*).	A solution to this problem can be to send random samples batch wise for study in the lab for a month or so.

Other Gemba observations –

- The PHE's present as a part of the heating circuit still leaks minor amount of liquid.
- The pulper still leaks minor amount of NMMO around the gland.
- The current display in the 0.5 TPD pulper section is still non-functional. The primary utility of this display is that it helps the operator to understand if the pulper is proper in operation.

As already mentioned the Melt Flow Index (MFI) is one of the most critical parameters characterizing dope quality. MFI is a unit of viscosity. If viscosity is very high then the dope will lose its flow properties and will thus increase CPF I/L Pressure while if the viscosity of dope is less than the desired consistency then it cannot be spun properly. **The primary reasons for the MFI variation and some suggestions for improvement are tabulated below-**

Table 3.2: Reasons for MFI variation and some suggestion for improvement

1. Variation of Hopper (Slurry) temperature. This temperature affects the LIST Zone 1 temperature where the major evaporation takes place (*refer plot below*). The hopper temperature is impacted by the NMMO line temperature which was confirmed during the sampling study conducted in the 0.5 TPD pulper section	A PHE can be installed to ensure constant temperature of NMMO sent to the pulper for each batch preparation. If this is done then one parameter can be eliminated and we can move further down the process for other corrective actions.
2. Variation in LIST Zone 1,2 temperature	Ensure constant slurry temperature in the hopper which affects Zone 1 and which in turn affects Zone 2 temperature

3. Erratic DKD cycle time (*refer plot below*) which results in varying amount of slurry entering the LIST	The control of DKD is manual in nature and is varied when the hydraulic pressure increases/decreases. Now Hopper Temperature affects Hydraulic Pressure (refer plot below) and this in turn induces change in DKD Cycle time. So Slurry temperature in the hopper should be maintained.
5. As per observation, the pulp consistency in terms of cellulose % and moisture content is questionable and should be looked into as it directly affects MFI (*refer plot below*).	A solution to this problem can be to send random samples batch wise for study in the lab for a month or so.
6. Discharge Zone and Shaft temperature varies continually. Discharge Zone temperature is varied to maintain Zone 4 temperature which is affected by Zone 3 temperature which is in turn indirectly affected by Hopper temperature	Maintain Hopper temperature to maintain the other parameters.
7. The sampling interval irregularity (*discussed below*) makes it difficult for data comparison.	Proper schedule for sampling have been proposed below

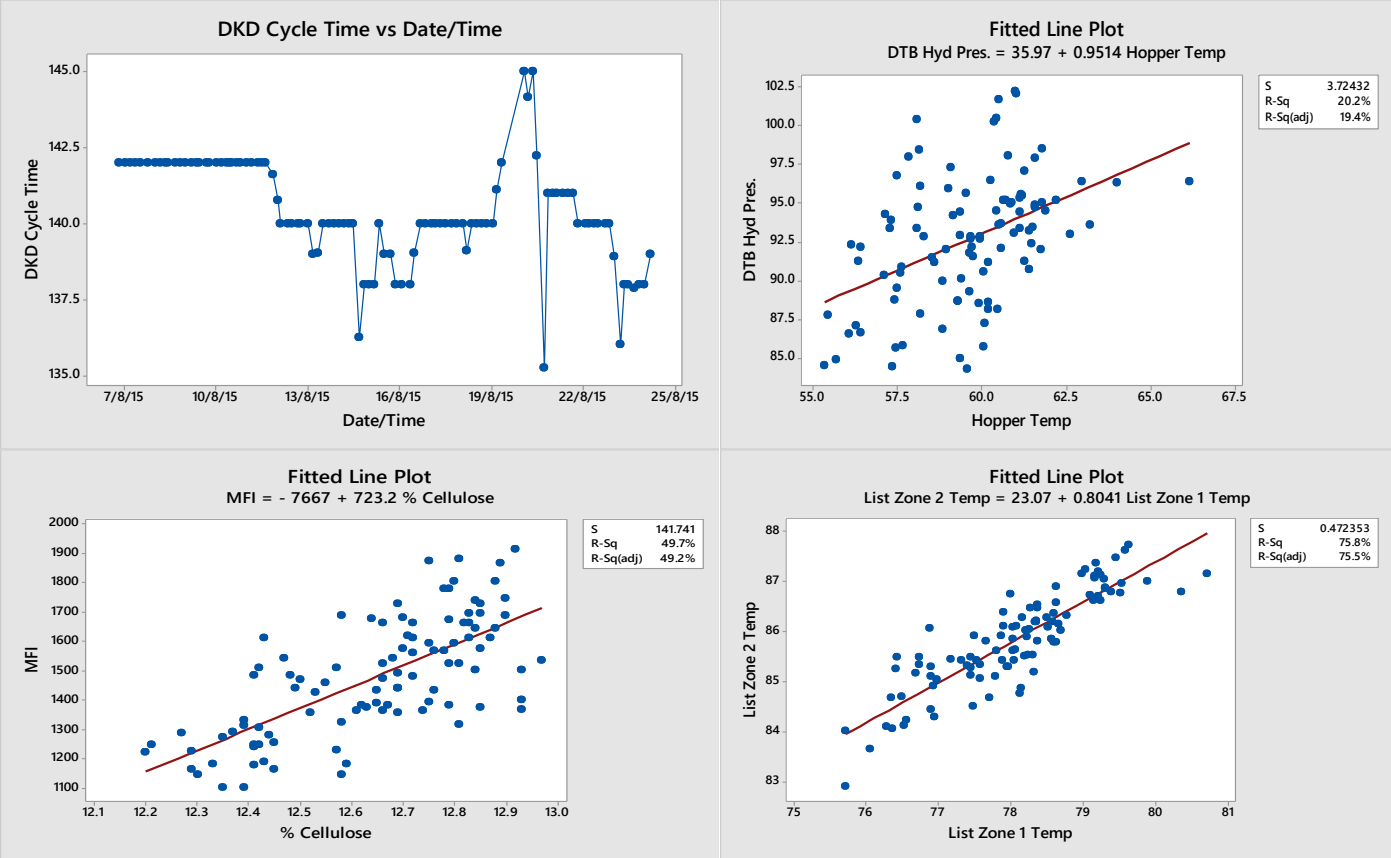


Table 3.2 and above plots gives us significant correlations between Hopper temperature and LIST parameters which varies the dope quality. All of the corrective actions suggested can only be a hypothesis now as most of the parameters are varying and it is very difficult to ascertain the necessary action. It is more like a trial & error and process of elimination kind of task. We have to apply corrective actions sequentially from the starting of the process and keep on eliminating the parameters based on effect of corrective actions.

3.5 Influence of Hopper temperature on other parameters:

The slurry temperature in the pulper after the batch preparation is around 85-95 ^{0}C while the slurry in the hopper prior to batch dumping has a top surface temperature of around 72-78 ^{0}C. The above mentioned temperatures have been manually measured by mercury thermometer. A RTD measuring the temperature of the slurry is installed at the bottom of the hopper which gave us a reading in the range 55-66 ^{0}C which is fluctuating in nature. There is a difference in temperature of around 15-20 ^{0}C between the top and bottom layers of slurry which is pretty high considering the fact that the hopper is jacketed with a circulation of hot water having a

set point of 80 ^{0}C. It is suggested that this temperature must be maintained for desiring constant parameters in the LIST because constant slurry temperature will ensure minimum temperature variation of the heating circuits in and around LIST and should result in constant evaporation. Relation between Hopper Temperature and other parameters have been studied and suitable fitted-line plots have been drawn to give us a better picture of the problem.

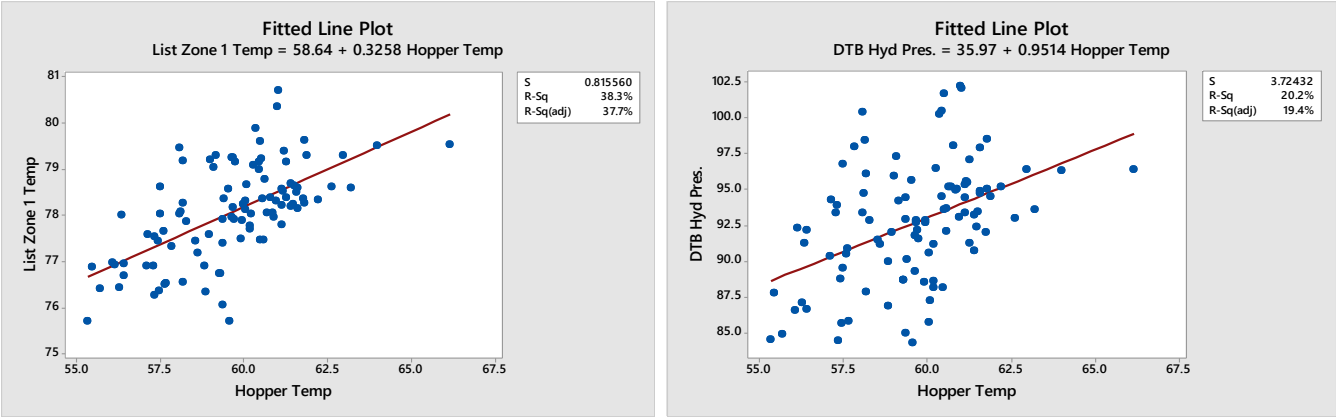


Fig: Fitted Line plot between LIST Zone 1 Temperature and Hopper Temperature, DTB Hyd. Pressure and Hopper Temperature.

From the above plot it is evident that Hopper Temperature strongly influences LIST Zone 1 temperature and weakly influences DTB Hydraulic Pressure. The Zone 1 temperature in turn influences Zone 2 temperature and Zone 3 temperature affects Zone 4 temperature.

Statistically proving this relation we have,

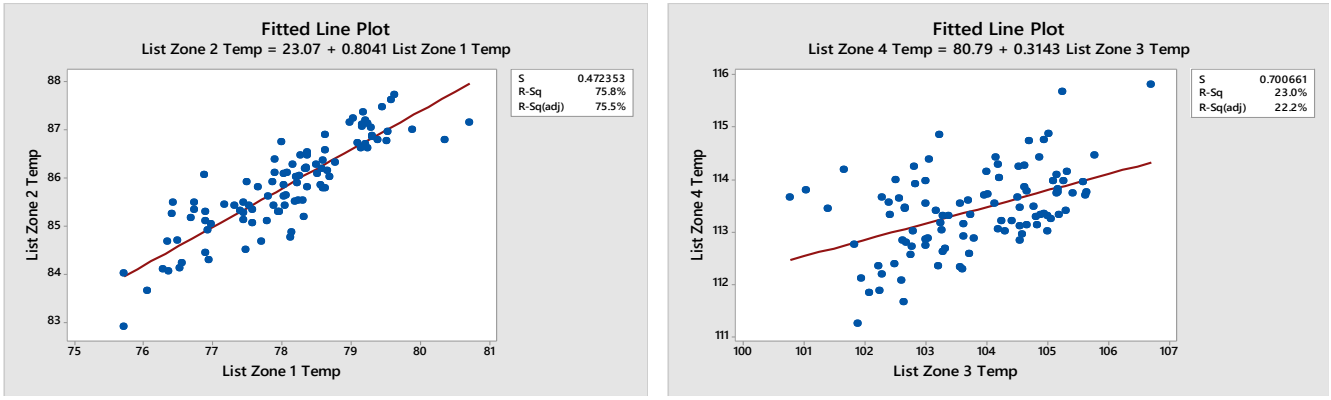


Fig: Fitted Line plot between LIST Zone 1 Temperature and List Zone 2 Temperature, List Zone 3 Temperature and List Zone 4 Temperature.

The major area of concern is the significant amount of heat loss which I have already mentioned above. So the reading given by the RTD installed at the bottom of the hopper might be misleading.

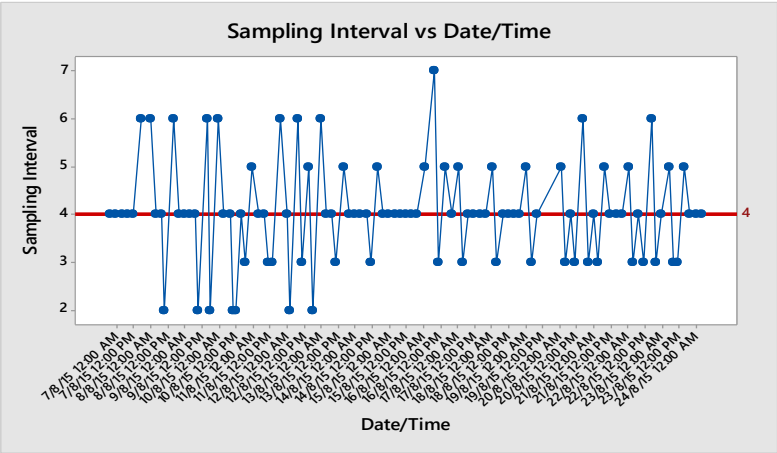
3.6 MFI sampling interval irregularity in regular process:

The polymer sample analysis in the lab primarily for detecting cellulose%, MFI, moisture content, NMMO % etc. is done as 2 samples per shift i.e. six samples per 24 hours.

Perfect schedule should be followed for polymer sampling to have a better idea about the variations. If random checking is necessary, then other timing can be adhered to but not at the cost of the original schedule. The other time can be followed in addition to the normal schedule.

During my study of the 0.5 TPD plant I observed that there is no planned time for sample collection and the intervals between sample collections is very erratic as seen in the plot below. A sampling schedule having a uniform interval of 4 hours is suggested below-

Proper sampling Schedule
8.00 am
12.00 pm
4.00 pm
8.00 pm
12.00 am
4.00 am
8.00 am



Proper interval should always be considered while collection of sample as this gives complete time for slurry to dope preparation. Scheduling sampling also ensures convenient study and better comparison of data. The **plot** attached above shows high variations (irregular time intervals). The red line indicates the proper interval of 4 hours which should be followed. The above mentioned sampling time can be adhered to as it gives uniform spacing (4 hours) and also provides the lab personnel ample time for completing the previous dope analysis.

4. STUDY OF 10 TPD COMMERCIAL PLANT

4.1 Process Description with basic process flow diagram:

4.1.1 Pulper

The pulping operation is a batch process contrary to the whole Excel fiber manufacturing plant which is continuous in operation. 120-130 kg of low viscosity (Cp) cellulose sheets depending on cellulose percentage in dope are weighed for 10 TPD production and kept ready to be sent to the pulper. An overhead tank containing 64-65% concentrated N-methylmorpholine N-oxide (NMMO) is also kept ready. That is the water percentage is around 35-36 %. The overhead tank is filled up to 520 mm or till the view glass installed in the overflow line shows runaway NMMO. There are two pulpers present and are alternate in operation. For running one of the pulper's firstly the dump valve is closed which ensures closure of the exit of the pulper. Then via a pulley mechanism cellulose sheets are added to the pulper. Firstly 40-50% of the cellulose sheets are added. Then NMMO drain valve is opened to facilitate its flow to the pulper. The pulper consists of an agitator which ensures breaking of the sheets and homogeneous mixing with NMMO. The pulper facilitates the solvation of NMMO with cellulose where hydrogen bonds are formed among cellulose, NMMO, water. For a fresh batch of pulp, the pulping operation is run for 30 minutes. Then the dump valve is reopened and the slurry formed is then sent to the hopper. The hopper is heated by circulation of hot water. The slurry will be sent to the hopper only if the level of the hopper is below a defined level. Now, the slurry is sent to WFE using a slurry feed pump.

The PFD is given below-

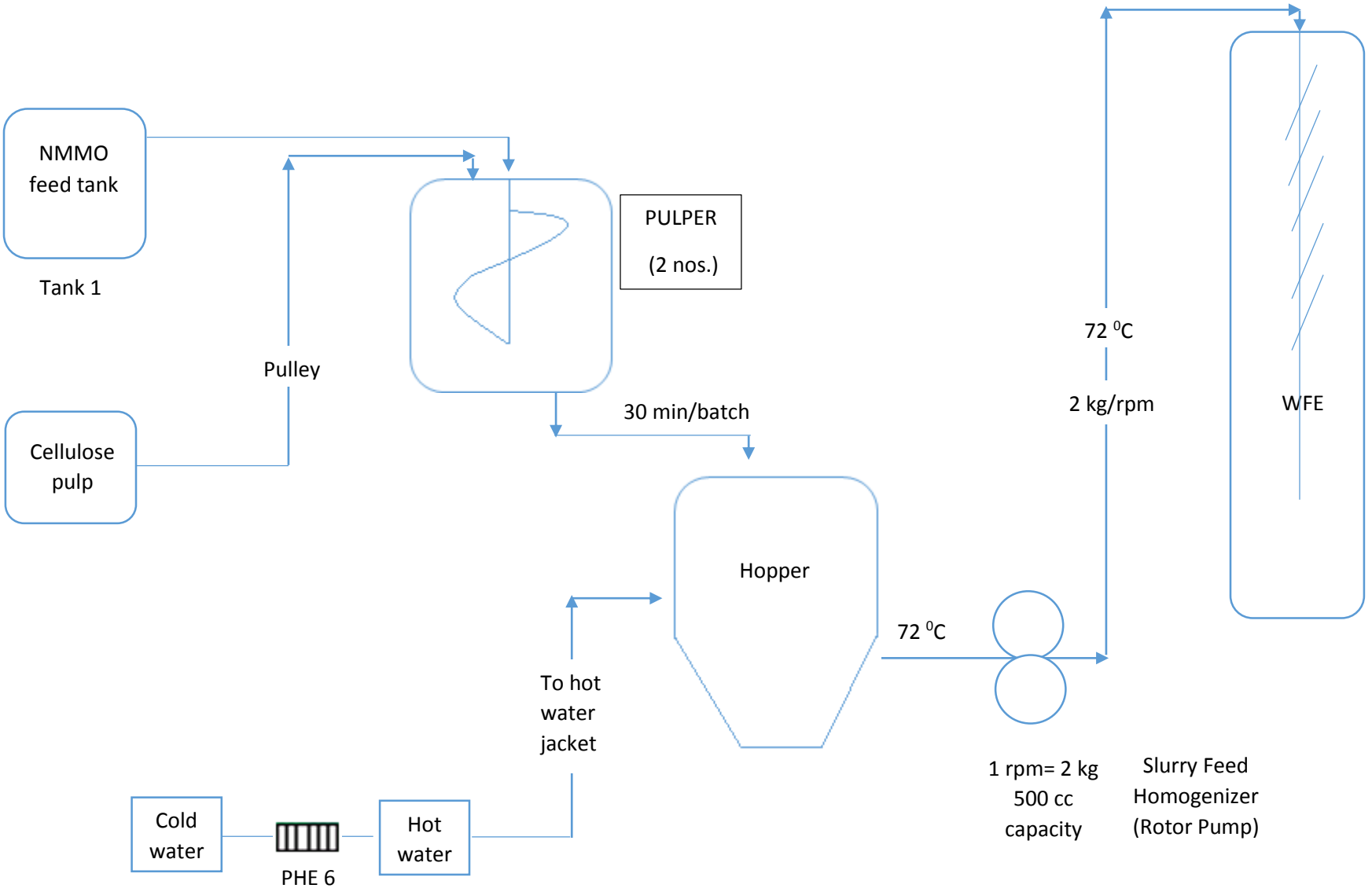


Figure 4.1: Pulping Section

4.1.2 Wiped Film Evaporator

The working of the wiped film evaporator is one of the most important part of the Excel fiber manufacturing plant. For the 10 TPD plant the WFE is divided into 4 shells each externally steam jacketed. There is no division internally as such. The preheated slurry at 72-80 ^{0} C coming from the hopper is sent to the WFE via a progressive cavity pump running at certain rpm where 1 revolution of the pump is equivalent to a throughput of 2 kg slurry. The slurry entering through the top of the WFE meets the rotor which is rotated using a hydraulic motor employing a pressure of 160 bar. This rotor having blades facilitates the homogeneous formation of the film. The film thus formed starts moving down because of rotor action and its blade angle. The clearance between the rotor and the shell is about 3-5 mm. The whole unit is maintained under vacuum. The purpose of vacuum creation is to facilitate the evaporation at lower temperature. The corresponding shell temperature ranges are given as under-

Shell I- 135-131^{0}C

Shell II- 134-130 ^{0}C

Shell III- 133-129 ^{0}C

Shell IV- 132-128 ^{0}C

These temperature are varying in nature but the difference between each shell is generally about a degree or two.

The steam used in jacketing the WFE shells is HP steam sent from the header. The steam after giving out its heat for evaporation is passed through steam traps. The condensate formed is passed out while the remaining steam is recirculated. This condensate formed is recovered and used for generation of LP steam. The removal of vapors generated in the WFE is quite interesting. The vapors generated is sent through vapor line. This is then sent to a collection drum where NMMO gets collected as condensate. The stream is then sent to the primary condenser. The condensed water is then sent to the hot well. From the hot well, water is sent to water pond where make up water is also added. Water from the water pond is sent to the primary condenser at the top via a U-seal to condense the hot vapors coming from WFE. This U-seal is present to maintain minimum level of water. The vapor line is attached to the primary condenser near the bottom. The water flowing inside the primary condenser from the top carries the vapor along with it. This is the primary source of vacuum and creates pressure up

to - 448 mm Hg vacuum. The remaining non-condensable are sent to a two stage steam ejector controlled by a control valve which is air to close type. These ejectors then bring the pressure to -658 mm Hg vacuum required inside the WFE unit. The vapor still remaining is vented out to the atmosphere.

The major part of evaporation takes place in the first two shells and brings the ratio of NMMO and water to 76:12. Due to this degradation and dehydration the slurry now called dope looks reddish in color. The degree of polymerization also decreases. Anti-oxidants are added to prevent oxidation and corrosion. The desired viscosity of this dope is 700-800 poise and refractive index of 1.4840-1.4850. The dope coming down after Shell IV meets with a scrapper kind of rotating equipment which scraps out the dope onto the cone. The dope is then pushed forward by a discharge twin screw conveyer. This is then moved forward by the WFE booster pump. Then this stream is divided into two streams A and B and sent to the filter pump. All the stream lines coming from the cone are heat traced at 106^0 C -108^0 C depending on the percentage of cellulose. This is done in order to maintain the flow properties of dope. The water utilized to heat the assemblies is heated by Plate heat exchangers employing steam. The PFD for the Wiped Film Evaporator section is given in the next page.

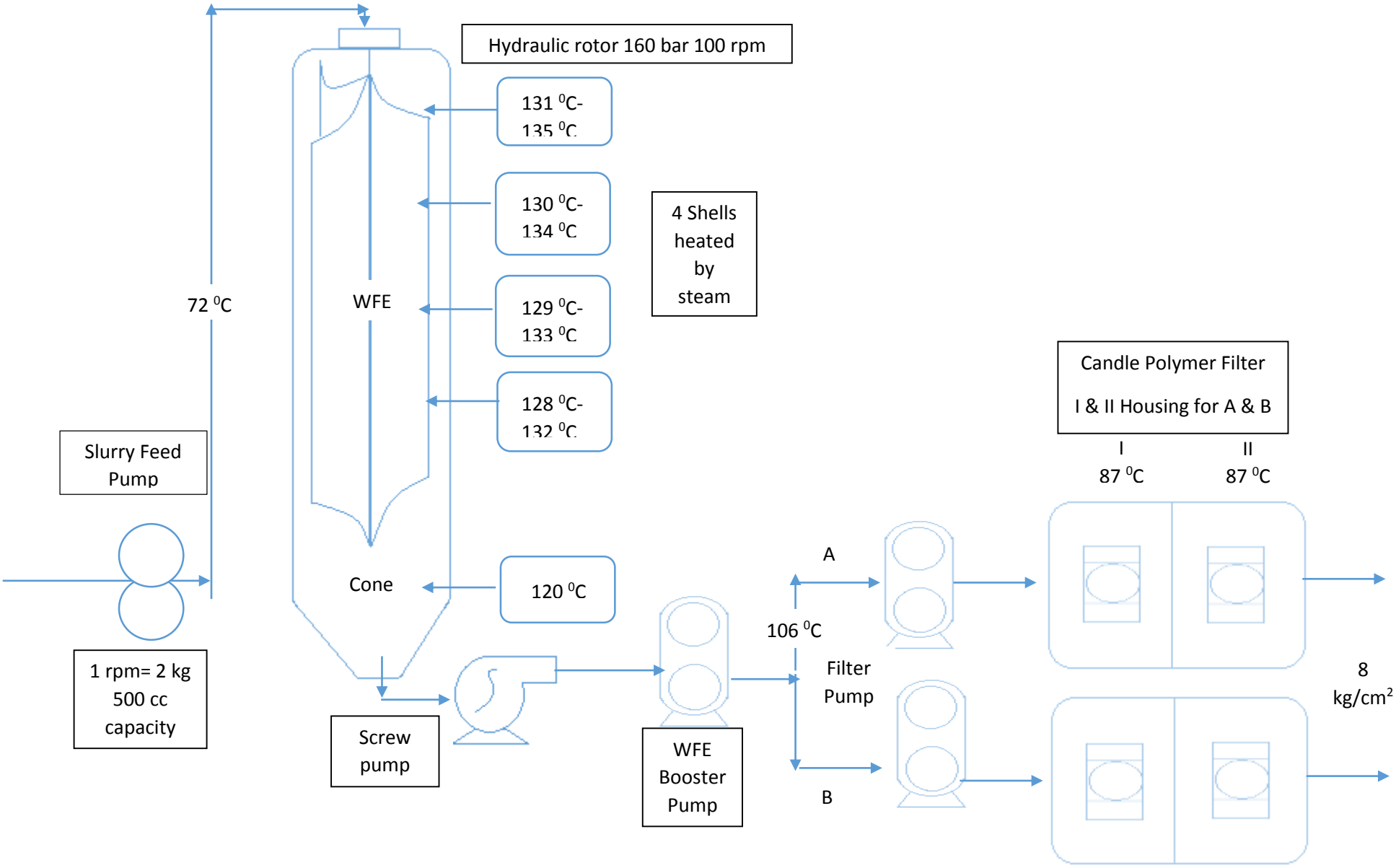


Figure 4.2: WFE and CPF Section

4.1.3 Spinning section

The stream coming from the candle polymer filter (CPF) gets divided into three headers. One of the modules present has two lines. The dope are pumped by the gear pumps running at around certain rpm. The total rpm of all the pumps is around 350 and depends on the process requirement. Suppose there is a drop in the hydraulic pressure of the WFE, then the rpm of the spinneret is reduced so that more dope can be accumulated in the cone section of the WFE. This in turn actuates the rotor to build up more pressure to homogenously spread the slurry. The dope enters the spinnerets by the inlet in the jet module. A hot air line is attached from the top where air from the Air Handling Unit (AHU) is blown at high speed to facilitate the angular moving out of the fiber strands. The spinneret is installed over an NMMO dilute solution open tank like arrangement called gutter. This NMMO solution is maintained at a concentration of 24-25 % using the dope which contains NMMO flowing in from the modules. There is an air gap of 20-22 mm between the spinneret and the solution level. The spinning occurs in two stages- Dry spinning, Wet spinning. The spinning which takes place above the solution level is called Dry spinning while the spinning taking place below the solution level is called Wet spinning. Air quenching takes place in the process. The air gap maintained in the spinnerets facilitates the quenching process. When fiber strands come out, there is sudden cooling by the air passing. The regeneration of cellulose takes place in the wash. The fiber strands are then entangled together to form a tow and made to pass through different cylinder like arrangements via a guide where the fibers are towed to the rollers. While getting towed the fibers are rewashed with a dilute solution of NMMO (8% concentration). These processes are done in order to recover the maximum amount of NMMO. The fibers that are towed are then passed through the 1st squeeze roller where the excess solution is squeezed out. The fiber strands are now passed through the second tow wash tank where the wash water has 4-5% of NMMO. This stream is then passed onto the 2nd squeeze roller via a stretch roller which stretches the fiber to provide tensile strength. The fiber strands are then passed through a cutter feed roller into a cutter. A wash stream from cutter recirculation is run continuously to ensure moisture presence in the fiber strands before cutting. The cutter cuts the fiber strands into small segments. These are then sent to the after-treatment section. There are two assemblies based on lines A and B coming from the CPF. Both the assemblies are run at once till the second roller. Only one of the cutters is kept online while the other cutter is kept on standby. This is

done to ensure the continuity of the operation that might be jeopardized because of ripening of the fiber strands over the tow.

The basic methodology employed for maintaining the concentration of streams in the Spinning section and After-treatment section is recirculation of stream from succeeding segment to the preceding one.

The spinneret is made of different components arranged sequentially in the order (Top to Bottom)-

i. Dope in and Hot water in and out section
ii. 1st Distributor plate
iii. 35 micron filter media
iv. 2nd distributor plate
v. 40 micron filter media
vi. Spinning jet
vii. Air distributor plate
viii. Inside Body

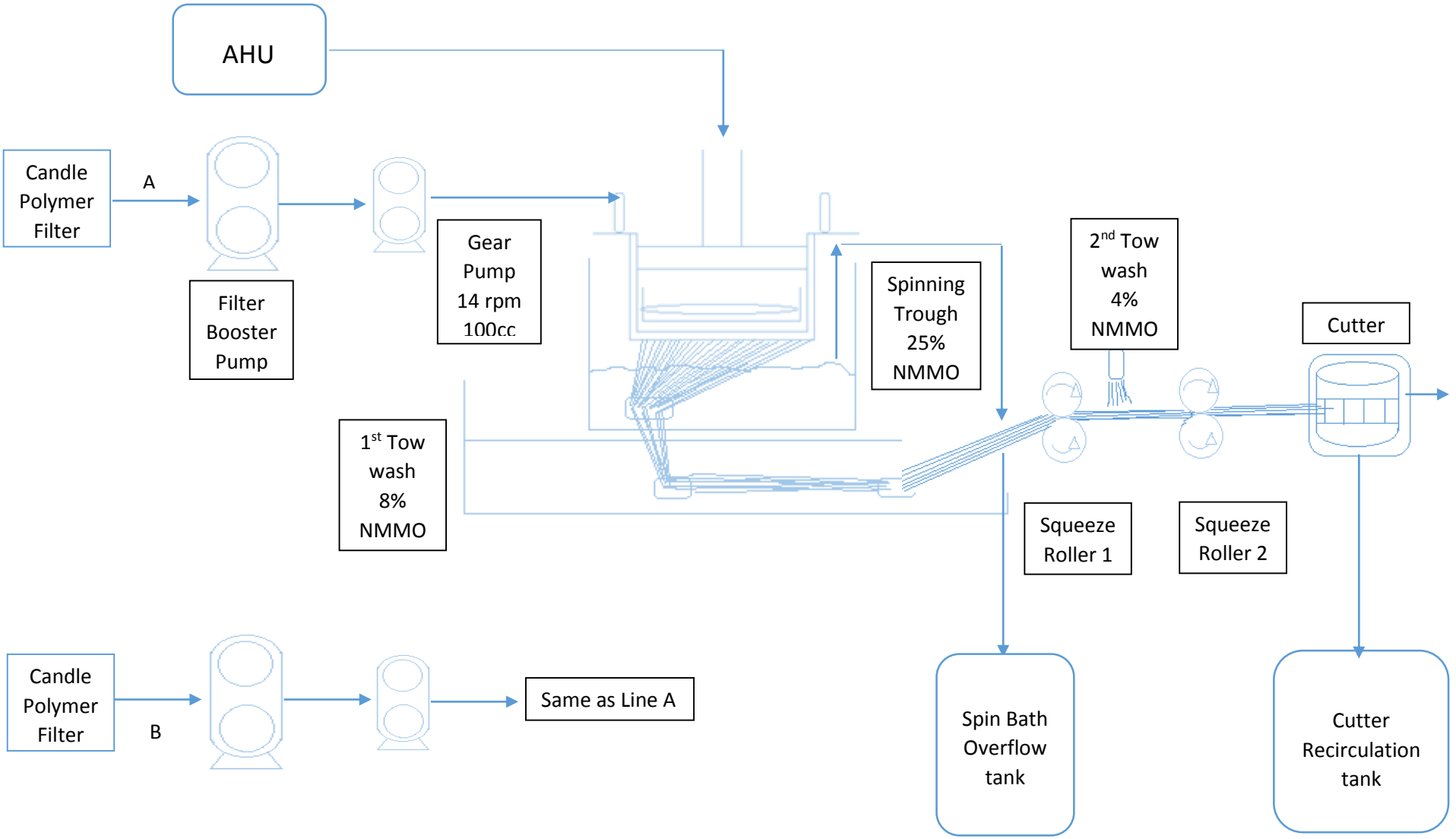


Figure 4.3: Spinning Section

4.1.4 After-Treatment section

The fiber after the cutter is sent to the after-treatment section. The after-treatment section is divided into various steps which are explained sequentially below-

a. Recovery trough- The fiber is exposed to LP steam at 0.5 kg/cm^2 which is done in order to blow out the water present and to open up the strands. The steam is distributed into pipes and passed through holes at an angle of 70-75 ^{0}. The fibers are placed over fixed bars which are moved forward by hopping action of moving bars. The water + NMMO gets drained by the screens vibrating and jerking forward. This motion facilitates the fiber to move forward to the NMMO wash tank. The water drained goes back as cutter recycle and circulation.

b. NMMO Wash tank- The fibers in this step are washed with water which comes from the cutter. This process is done to reduce the NMMO concentration in the fibers.

c. Squeeze roller 1- In this step simple squeezing operation takes place with the aid of rollers moving in alternate direction. This is a part of main drive 1.

d. NMMO Wash Tank 2- In this step the fibers are rewashed with stream coming from recovery.

e. Squeeze roller 2- In this step simple squeezing operation takes place with the aid of rollers moving in alternate direction. This is a part of main drive 2.

f. Bleach bath- Here the fiber strands are bleached to remove the reddish color and provide the whitish complexion. The bleach used is hypochlorite. Frequent dosing is done from a tank containing hypo to maintain the hypo concentration inside the bleach bath.

g. Bleach wash zone- Here the fiber is washed with water recycled from next wash zone.

h. Squeeze roller 3- In this step simple squeezing operation takes place with the aid of rollers moving in alternate direction. This is a part of main drive 3.

i. Wash Zone- Here washing of the fiber takes place with soft water to remove traces of bleach and NMMO. The water after washing is recirculated to the previous bleach wash zone.

j. Squeeze roller 4- In this step simple squeezing operation takes place with the aid of rollers moving in alternate direction. This is a part of main drive 4.

k. Soap wash Zone- Here soap and oil is added to provide the desired texture and softness to the fiber. The soap mixture solution is doped to maintain the concentration of the soap in the soap bath. This process takes place at 50-52^0 C.

l. Squeeze roller 5- In this step simple squeezing operation takes place with the aid of rollers moving in alternate direction. This is a part of main drive 5.

m. Nip roller- The objective of this equipment is to press the fiber and send it to the Mat opener segment.

n. Mat Opener- The Mat Opener is an equipment having teeth like arrangement which breaks/tears the fiber.

The fiber is then sent to the drying section.

The basic methodology employed for maintaining the concentration of streams in the Spinning section and After-treatment section is recirculation of stream from succeeding segment to the preceding one.

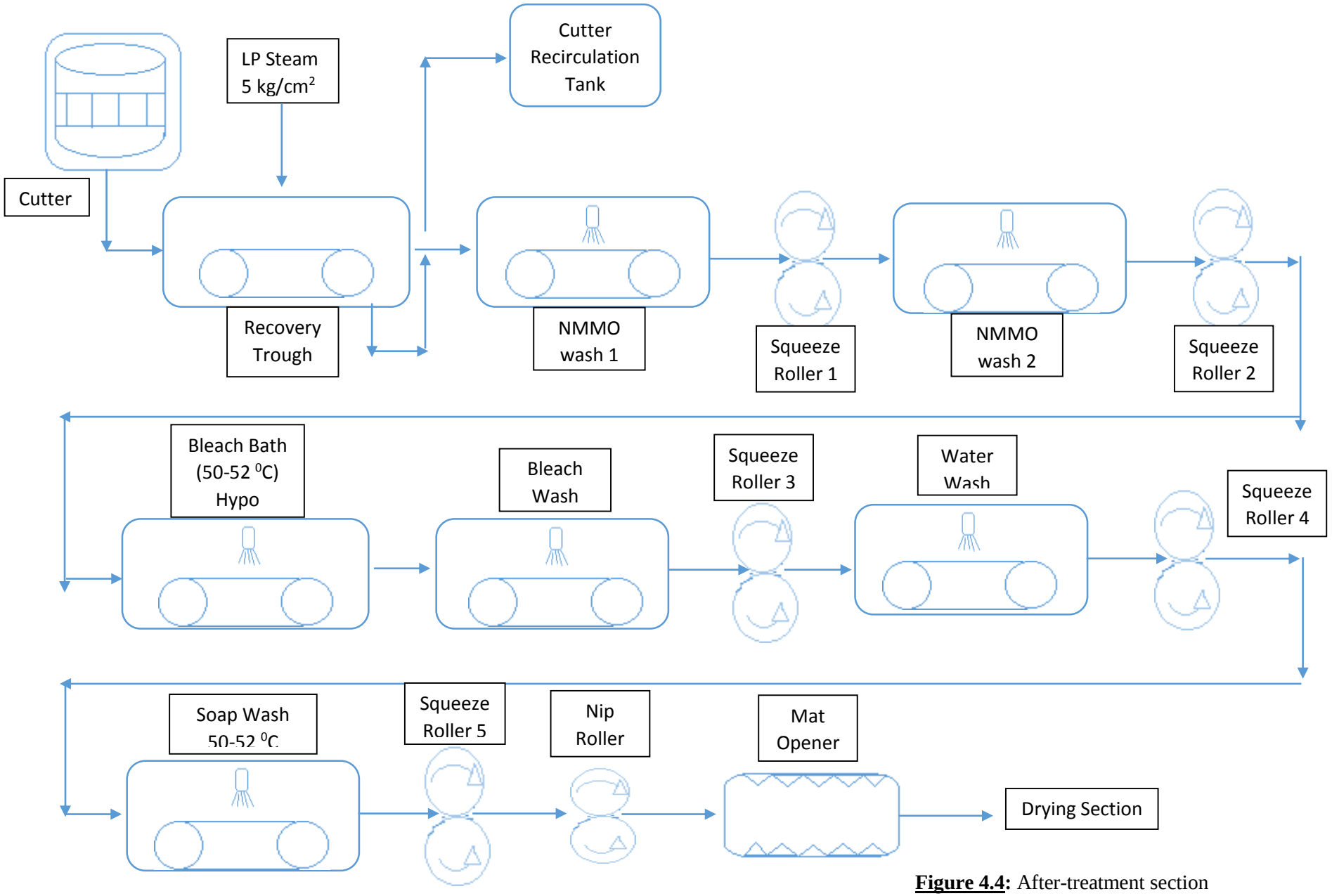


Figure 4.4: After-treatment section

4.1.5 Drying section

There are two dryer assemblies present. The 1st dryer assembly present has 7 coils while the 2nd dryer assembly present has 6 coils. Steam from the steam headers is divided into each assembly. The moisture in the fiber at the end of the 2nd dryer assembly is to be maintained between 9-11%. There are three nip roller and mat opener arrangements present. The first one is just before the drying section while the second is located after the 1st dryer assembly and is called the intermediate. The third one is after the 2nd dryer assembly. The steam enters from the top while air is blown by a blower from the bottom. The steam enters the coils and air is circulated over them. The hot coil heats up the air which facilitates drying. The entry percentage of the moisture before the 1st dryer assembly is about 110-130 % which reduces to 60-70% after the first dryer and then reduces to about 10-12% after the second dryer assembly. The fibers come out of the second dryer at around 40^0C. The drying section is divided into 4 zones.

Zone 1- 90-120^0 C

Zone 2- 100-120 ^{0} C

Zone 3- 110-130 ^{0} C

Zone 4- 90-110^0 C

The fiber after drying is then sent to the baling press where compression is done under 80-90 kg/cm^2. The saturated air is vented out.

4.1.6 Baling Press section

The fiber after drying is sent to the baling press where compression is done under 80 kg/cm^2 pressure. Here in this section Bales are made each weighing around 254-255 kg. The bales made are passed through a Forte system. This is a unique system which superficially divides the bale into sixteen segments. This system then analyses the moisture present in each of the segments. The overall permissible moisture in the bale is 9-11% while the permissible moisture in the individual segments is 8-13%. If too many of this segmental moisture value is exceeded then the bale is broken down and resent to the 2nd dryer assembly to maintain the moisture level.

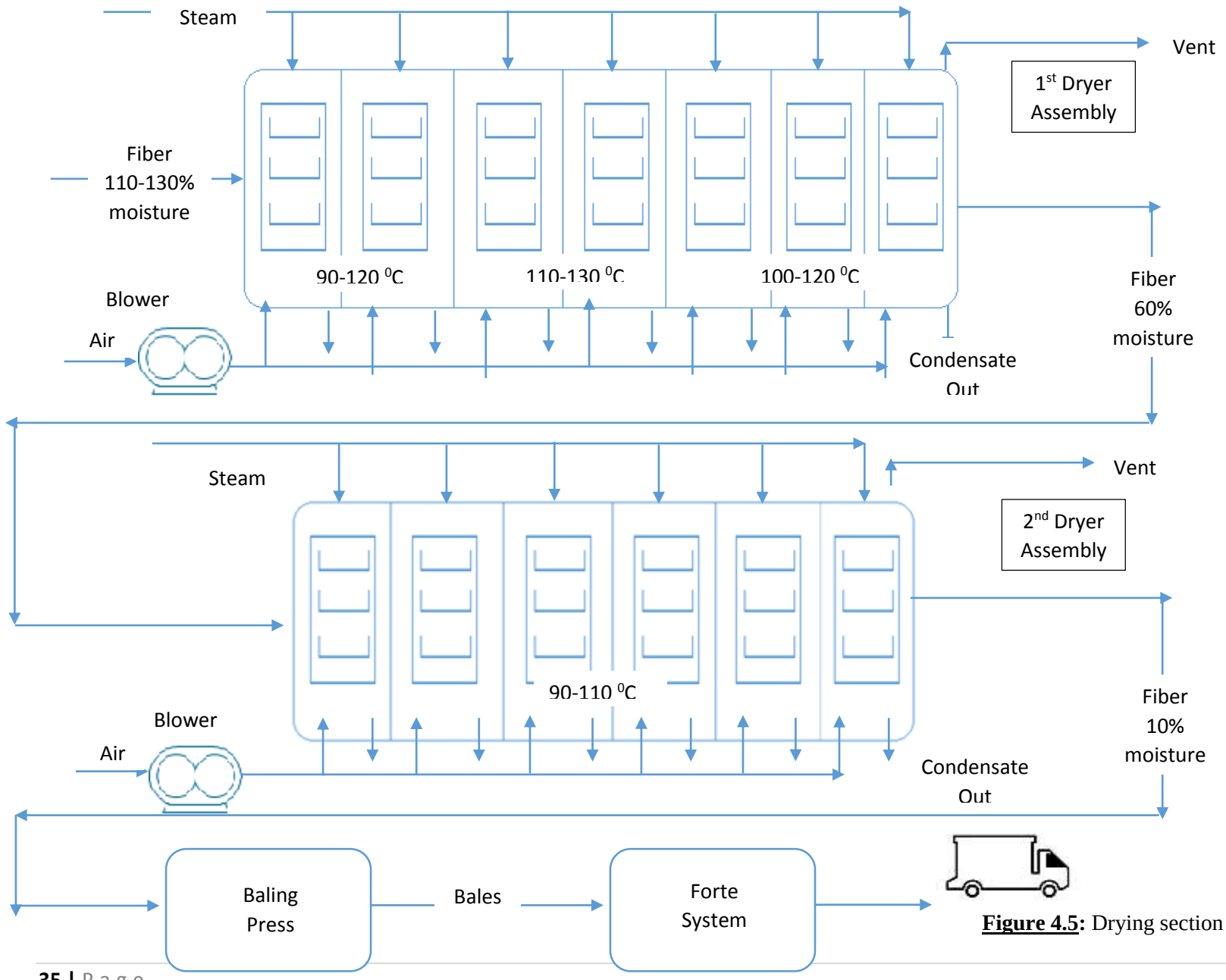


Figure 4.5: Drying section

4.2 Sampling exercise in pulper section:

4.2.1 Work done in the exercise

The sampling exercise comprised of taking down parameters important for the process. The 10 TPD plant is experiencing variations in the process parameters. Certain process parameters are out of their range for significant amount of time. The 10 TPD plant consists of several sections of which the pulper was first selected for study as this section is responsible for processing the raw materials and thus primarily deciding the quality of fibers. The objective of this section-wise study (Sampling Exercise) was to identify the causes due to which the parameters are varying. The sampling exercise was more like a process of elimination. The problems from a section can be identified and rectified and that section can be crossed off from the list and the following section can be studied.

In the pulper, cellulose and NMMO are mixed to form a slurry which is then sent to the WFE. There are two pulpers which are alternate in operation. The mixing time allowed is 30 minutes for each batch preparation.

The pulper itself can be sectionalized as-

i. Pulp weighing machine
ii. NMMO overhead tank
iii. Pulper to form slurry (2 nos.)
iv. Hopper
v. Slurry feed pump

The targeted parameters were-

i. Weight of the pulp taken
ii. NMMO temperature sent to pulper
iii. Slurry temperature
iv. NMMO Refractive Index
v. NMMO conductivity
vi. Slurry sampling from pulper and slurry feed pump for Yield stress measurement

Task entrusted to me–

- To detect problems in the pulper section and propose solutions
- Noting weight of pulp taken for each batch
- Collecting a piece of pulp from every batch for its viscosity calculation
- Noting NMMO temperature in overhead tank
- Noting Slurry temperature
- Collecting slurry from each pulper every two hours for study.

The weight of pulp for each batch was noted down. The pulp used was of low viscosity (11-12 Cp). In order to validate this, pieces from pulp sheets were collected and sent to the lab for examination.

Next the temperature of NMMO from the overhead tank was noted down by collecting NMMO in a bottle from the sampling valve. This value gave us the idea about the temperature of NMMO at which it enters the pulper.

There is a timer installed in the pulper control which automatically stops the agitator after 30 minutes. This ensures that there is constant mixing time for every batch. After the agitator was stopped, the slurry temperature was noted down for every batch. Slurry was collected from each pulper after every two hours and sent to the lab for testing. The discussion pertaining to this exercise is carried out in the subsequent pages.

4.2.2 Results of the sampling study

There were many batches of data taken so only the ranges are specified.

- Pulp weight- 129- 129.1 kg
- NMMO temperature- 48- 74 0C
- Slurry Temperature- 78- 89 0C
- NMMO level- 520 mm (constant)

The NMMO temperature is particularly important as the pulping operation is exothermic. So in order to achieve a uniform slurry temperature, this temperature must be maintained constant for every batch. The NMMO and the slurry temperature were measured manually by a mercury

thermometer. The distance between 'Feed to pulper tank' and NMMO overhead tank is about 100-150 meters. The line carrying NMMO between these tanks is insulated. This line is exposed to the atmosphere and there might be a possibility that ambient atmosphere has an effect on the NMMO temperature. To avoid this a solution has been proposed in the next page.

I have identified some problems in 10 TPD pulper and have proposed solutions for the same which are tabulated as under.

Table 4.1: Problems found in 10 TPD pulper and their proposed solutions

Problem	Proposed Solutions
1. Grease from the hydraulic pulley system falls on the pulp during pulp entry into the pulper. More amount of grease can lead to tow darkening or can destabilize the process.	A barricade can be installed on sides which does not allow the pulp sheets to be displaced from its original position nor provide hindrance to the workman loading pulp sheets
2. NMMO sampling valve leaks and leads to loss of NMMO	The spindle or the washer should be changed in order to stop the leakage
3. NMMO leakage in line from pulper to hopper. This leads to reduction in quantity of NMMO in the slurry	Instead of wrapping a cloth around the then thought inconspicuous hole in the pipe, it should be fixed with heat resistant epoxy putty which can easily hold on to metals up to temperatures around 700 ^{0}C
4. Pulper does not become empty even after dumping. This leads to accumulation of slurry and formation of hot pockets on the agitator. This might affect the quality of fibers	The pulper can be flushed with water at high velocity. For this we require a small pump with a high discharge velocity and a garden hose connected to a drum having water. A small stream of water will cut through the layers of slurry stuck on the agitator and the walls of the pulper and will thus facilitate the cleaning. This should be done at regular frequencies. The washed out slurry can be sent for leaching.

5. The level sensor installed in the hopper gives different value at Pulper and WFE. I do not agree this to be a transmission lag as the value displayed at the pulper is lower contrary to the location of the sensor which is nearer to the pulper. The difference between the values is around 6 units. Different values can create confusion among the operators and can hamper their coordination. It can also lead to over/under dumping of slurry than required in the hopper.	A resolution of this problem can be to install wireless instrumentation which is slowly becoming the crux of instrumentation in industries today. Wireless instrumentation is more reliable and has greater impulse in comparison to local instrumentation as assessed by the manufactures.
6. Level sensor installed on the hopper gives fluctuating values. Fluctuating values creates confusion among the operators and influences their actions. It can lead to under dumping of slurry as per production in the hopper or it can lead to over dumping of slurry which in turn will exceed the maximum level of hopper.	According to the operators, the level sensor gives fluctuating values and this on increasing indicates the operators to clean the sensor. Cleaning is also done periodically but the problem recurs after 8^{th} or 9^{th} batch post cleaning. The best way for resolving this problem is to install a Laser level sensor which will give us non-contact & continuous level data.
7. There is an 'Agitator on' indication in the pulper panel box but according to the operators it sometimes gives false signal.	This problem should be rectified so that the operator can be assured that the agitator is on and need not check it using a torch.
8. Human error in weighing of pulp sheets.	This error can be altogether eliminated by manipulating the system. The weighing machine can be tweaked such that a minimum and maximum level can be set. The workman has to weigh pulp only in that range otherwise the hooter will keep on ringing. For 11.8% cellulose, the weighing machine range can be set as 129.00 to 129.01 kgs. The manual switch off button for the hooter has to be cut out for this purpose.

9. Variability of temperature of NMMO in the storage tank above the pulper. Maintaining a constant temperature of NMMO is extremely important as this influences the temperature of the slurry which in turn affects the entire process. Moreover RI and electrical conductivity is dependent on temperature and any change in temperature will also change the associated parameters. This will bring in difficulty in controlling the optimized values of RI and electrical conductivity and thus affect quality of fiber produced. The line supplying the NMMO from the tank is long and exposed to the atmosphere. Environmental factors also play a role in variation of NMMO temperature	Jacketing of NMMO storage tank above the pulper with a circulation of hot water using a PHE Or Building a storage tank in the vicinity of the pulper which will be jacketed with a circulation of hot water using a PHE. This will facilitate the maintenance of constant temperature of feed NMMO. Installation of an RTD indicating the NMMO temperature in the NMMO tank is also necessary.

4.3 Fish-Bone diagram identifying parameters affecting Melt Flow Index:

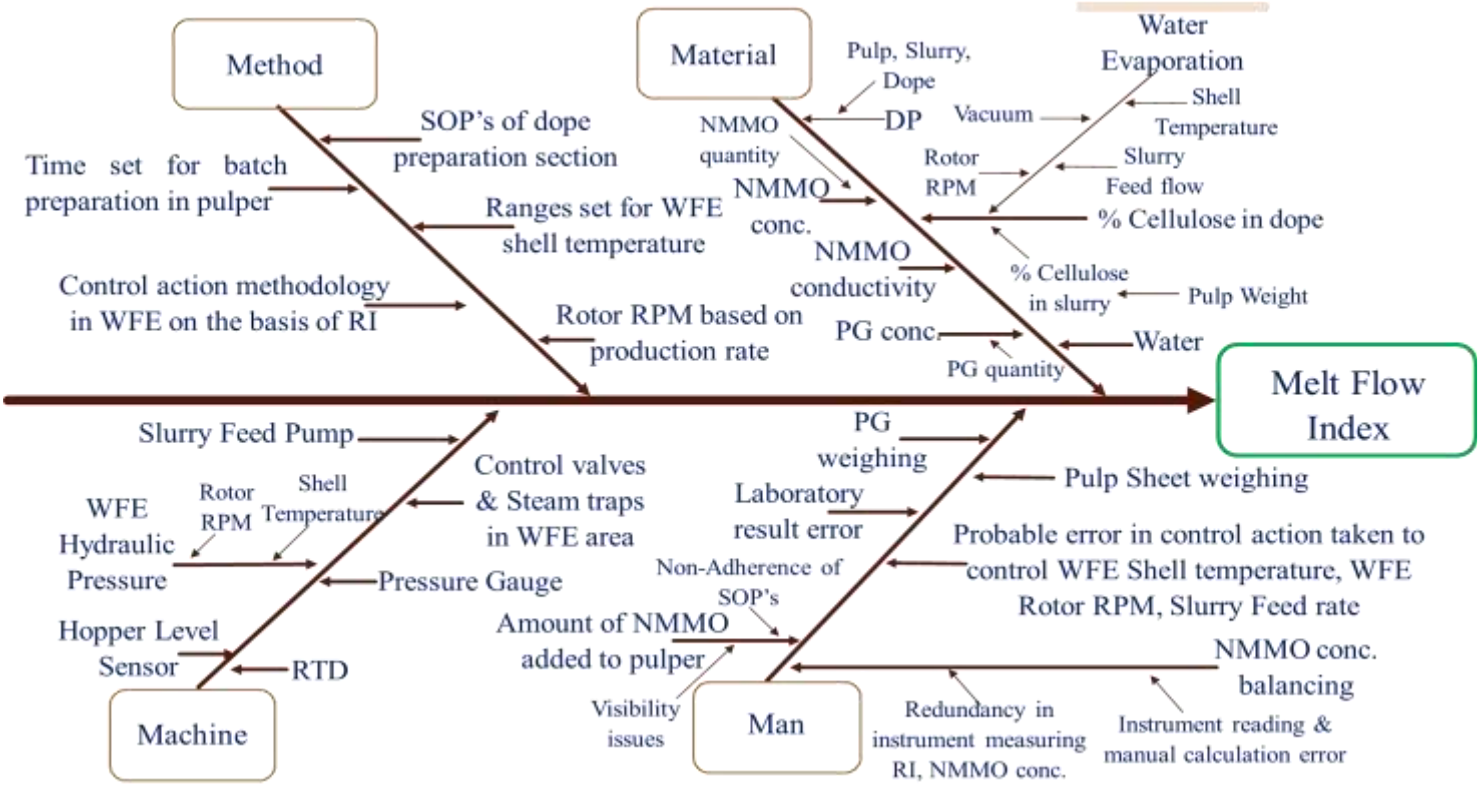


Figure 4.6: Fish-Bone diagram identifying parameters affecting Melt Flow Index

4.4 Theoretical relation between the identified parameters and Melt Flow Index:

NMMO temperature- NMMO temperature has a clear impact on slurry temperature since NMMO and cellulose pulp are the two basic raw materials entering the pulper. The pulper here in 10 TPD is itself not jacketed and there is no external heat supply to influence the process. Therefore NMMO temperature and the temperature evolved during the mixing process are the only sources of temperature to have any impact. If the NMMO temperature is high it will lead to higher slurry temperature. After the batch is prepared it is dumped into the hopper. The hopper is jacketed by hot water coming at 72 ^{0}C. Now if the temperature of the slurry is higher, it will transfer its heat to the jacket water and itself get cold. Now this slurry after getting cold might create problems in pumping or might affect the overall viscosity of dope as the evaporation temperature will be the same and so this dope will have further reduced moisture content.

NMMO concentration- If the NMMO concentration is higher than desired value of 64%, then the dope forming in normal evaporation conditions will have higher NMMO %. This will lead to increased RI. The general control methodology followed in the 10 TPD plant is on the basis of RI. If RI increases, the Shell temperatures are decreased. This affects viscosity. Moreover if the Shell temperature does not decrease RI, then Rotor RPM is decreased which also will affect MFI.

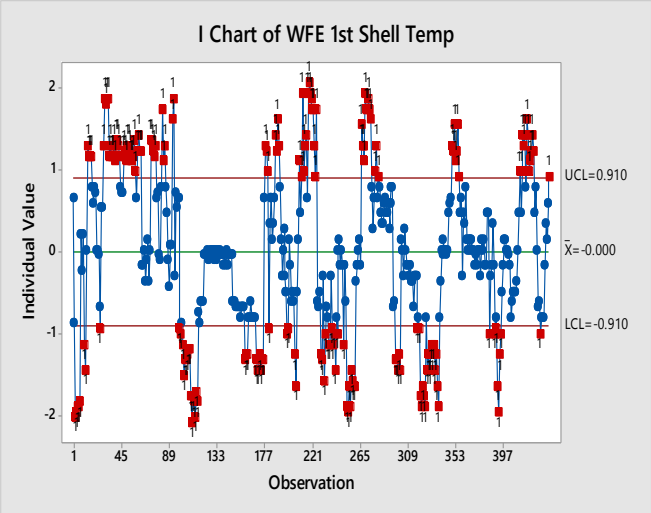
Pulp quantity- Pulp quantity is directly proportional to the % cellulose that is being added to the pulper and % cellulose directly affects Melt Flow Index.

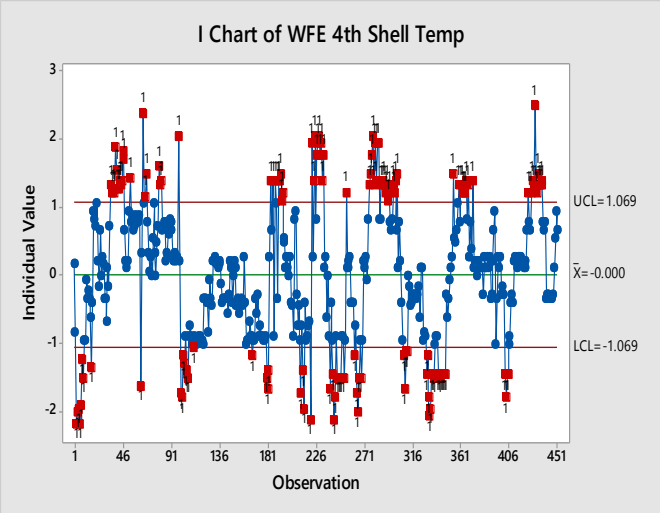
Pulping time- This is generally decided on the basis of quantity of pulp put in the pulper. The calculation must ensure that the pulp is properly dissolved in NMMO and made into slurry otherwise the process will be disturbed. If pulp does not dissolve properly then it might choke the pump, increase the hydraulic pressure thus putting a load on the rotor. Also the dope formed will not be of desired specification.

Rotor RPM- Rotor RPM is decided on the basis of production rate. When the rotor rpm increases, the mechanical energy gets converted to thermal energy which further aids in evaporation.

Vacuum- On vacuum not kept constant, the boiling point keeps on varying which leads to differential evaporation. This leads to different Melt Flow Index of the dope formed.

Shell Temperature- The Shell Temperature is currently seen to be continually varying from the graph attached.





The Shell temperature is generally varied manually to control the rise in RI. Whenever RI increases the shell temperature is reduced to keep the value of RI in range. The Shell temperature also varies automatically because of leakage of condensate through steam traps. If steam comes in contact with the condensate then the temperature of steam (Shell temperature) fluctuates as it starts condensing. Moreover there is an inherent issue. A trend has been observed that whenever there is an increase in the Ion Exchange I/L conductivity there is an increase in Ion Exchange O/L conductivity and subsequently considering a time lag of two hours, a change in RI is seen which induces a change in Shell temperature.

Slurry Feed Flow- Slurry feed flow if not kept constant means different amounts of slurry is being sent inside the Wiped Film evaporator. This actually affects several other parameters namely Rotor rpm, Hydraulic pressure, effective evaporation etc. The Rotor rpm has been decided on the basis of production rate whose inherent part is slurry feed rate. If the rotor rpm is to be changed then there will be an effect on Hydraulic pressure too. Also if the slurry feed rate varies then this will effect effective evaporation as differential amount of moisture will get evaporated.

PG quantity (PG concentration) - PG is added for fulfillment of two objective. The primary objective is that at higher temperatures there is a chance of runaway reaction between NMMO and cellulose. But PG decreases the chemical affinity between them. If the quantity of PG added is not proper then

it directly affects the conductivity of NMMO which in turn affects the RI and shell temperature eventually varying the Melt flow index.

Degree of Polymerization- The degree of polymerization needs to be studied to understand the extent of degradation happening in the three different stages viz. Pulp, slurry making process, dope formation process. The amount of degradation characterizes the tow darkening phenomena.

Human error and negligence-

a. Probable error in control action taken to control WFE Shell temperature, WFE Rotor RPM. Some instances were seen when the operator leisurely operated the panel. A citation of an instance can be provided that the RI was dropping below 1.4842. The basic control methodology adopted in this plant is that whenever RI increases/decreases below/above 1.4845 – 1.4850, the shell temperatures are tweaked to maintain RI in that range. But 2-3 times this incident happened and there was no action taken. When pointed out, only then the operator took action. By this time the MFI might have varied.
b. The importance of PG weight and pulp weight have already been discussed above. So it is extremely important that the right amount of PG and pulp is added every time.
c. Laboratory result have been found to have variations as seen from the log books.
d. Amount of NMMO added to the pulper is important as it affects the concentration of NMMO entering the pulper.
e. Instrumentation viz. pump, RTD's, Steam Traps, Control valves have to audited periodically as some of them were found to be leaking (steam control valves, steam traps) and providing wrong values (RI meter, 3^{rd} & 4^{th} shell RTD). The steam traps have worn out and needs to be replaced. The RI meter specifically needs to be audited frequently as the value on the DCS panel and the RI panel are different. When calibrated it works fine but then comes back to its original state. Moreover for significant amount of time the Historian recording for RI values were wrong (in the range of 1.4700).
f. The region around the pulper has poor visibility and this affects the operators mainly during checking of NMMO level while making batch in pulper.

4.5 Comparative study of results of Melt Flow Index:

4.5.1 Procedure for the experiment

A comparative exercise was performed to check the results of the Lab personnel with experiments conducted with the same sample by me. The procedure for the experiments are given below-

MFI (Dope) & % Cellulose in dope-

The dope coming in must be heated in oven for some minutes. Then the container is taken out of the oven. About 2 – 3 g of dope is weighed and kept on a glass plate inside the oven for about 5 minutes. Next the dope is taken and loaded inside the MFI testing unit. The unit is half filled then the die at the bottom is fixed. Then keep on loading and press when the dope can be seen rising from the top until the air comes out. Then start loading again until dope is seen rising again. Now the loading point is covered and kept for 6-7 minutes. Now put on the weight of 3360 g over it. The level indicator will move down and suddenly a burst of air comes out. Then the dope will again come down. From 7 – 12 gradations the time is calculated. This time (in seconds) multiplied by 8.48 gives us the viscosity in poise.

For % cellulose the glass plate is taken out and is pressurized from the top with another plate so that a thin transparent film is formed between them. Now carefully slide the top glass plate over the bottom plate and keep in the water basin for the film to come out of the plate. This layer (film) is then sent for washing. The washing is done for about 40 – 45 minutes. After washing, the film is dried for 1 hour at around 105 ^{0}C. This dried film is weighed and divided by the initial weight of dope taken. This value when multiplied by 100 gives the % cellulose in dope.

% Cellulose in slurry-

The slurry brought in is washed with hot water. About 30 g of slurry is taken and the washing takes place in a crucible connected to a vacuum pump. It is washed several times to remove the traces of NMMO. The water at the bottom of the crucible is removed by attaching the lower part to the appendage of vacuum pump protruding out. Then the white cake (pulp) remaining behind is dried at about 105 ^{0}C for about 2 hours. The dried cake (pulp) is then weighed and divided by initial weight of slurry taken and multiplied by 100 to get % cellulose in slurry.

4.5.2 Result from the experiment

Table: 4.2 Experimental results for Melt Flow Index and % Cellulose in dope: (Self and Lab Personnel)

Sr No.	Plant	Melt Flow Index (Self)	Melt Flow Index (LAB)	% Error in MFI (Self)	% Cellulose in dope (Self)	% Cellulose in dope (LAB)	% Error in % Cellulose (Self)
1	20 TPD	415	457	9.19	10.88%	10.98%	0.91
2	10 TPD	435	483	9.93	11.11%	11.06%	-0.45
3	10 TPD	508	510	0.39	11.18%	11.07%	-0.99
4	20 TPD	457	449	-1.78	10.82%	10.84%	0.18
5	Pilot Plant	NA	NA	NA	12.18%	12.10%	-0.66
6	Pilot Plant	NA	NA	NA	12.08%	12.01%	-0.58
7	20 TPD	520	558	6.81	10.90%	10.90%	0
8	10 TPD	500	526	4.94	11.14%	11.06%	-0.72
9	20 TPD	NA	NA	NA	10.93%	10.99%	0.54
10	10 TPD	NA	NA	NA	11.12%	11.11%	-0.09

Table 4.2 indicates the error % in comparison of % cellulose of dope & Melt Flow Index between self and lab. The error % was found to be negligible i.e. most of the time the results can be relied upon.

4.6 Experimentation procedure to determine Cp:

1. **Pulp**

Grind the pulp into sample bits using a corrugated steel surface. Take 1.6 g of slurry and add distilled water to it. Now add it to the crucible attached to the vacuum pump. The crucible is to be first covered with a filter cloth. Then on the vacuum pump. Keep rotating the crucible so that all of the water present is drained out. Now wash it with acetone two times following the same procedure. Now take the pulp film over a filter paper and press it slightly. Now remove the cloth and keep the film inside the oven which is at a temperature between 102-105 ^{0}C for 5 minutes. After exactly five minutes take the film out and crumble the filter paper over it so that moisture is not gained by the film. Now take 0.51 g of this pulp.

2. **Slurry cake**-

Weigh 5 g of slurry each in two separate beakers. Dissolve the slurry in hot water. Add the slurry sample in the crucible and on the vacuum pump. Wash the cake with about 1.5 Liters of hot water till the yellowish tinge disappears. Repeat the procedure for the other sample. Then keep the cake formed in the oven for 40 minutes for drying. Take 0.51 g of this sample after reheating in the oven for 10 minutes prior start of experimentation.

3. **Dope film**-

Take 0.51 g of dried film after crushing into tiny bits.

Procedure-

Take the dissolving tubes and arrange them according to their numbers. Insert rubber seals into each of the caps in order to prevent the spillage of the solution. Fill in 0.51 g of the respective samples into each tube. Then add steel beads to the tube. The number of steel beads to be added is according to sample. The dope film needs more number of steel beads to be added in comparison to pulp sample. Now add 50 ml of Cupraammonium solution to each of the dissolving tubes. Then add 0.15 ml of Pyrogallol solution (1 ml distilled water added to 0.5 g of Pyrogallol powder) to each of the tubes. Now seal the tubes tight and keep in the shaker for 35 minutes for pulp & slurry cake sample and 1 hour 30 minutes for dope sample. After shaking in the shaker take the tubes out and keep in the bath to normalize the temperature. Prior that ensure the temperature of the bath is 20 ^{0}C. If not add hot water and bring the temperature of the bath to 20 ^{0}C. Now take each tube orderly, shake it properly and then pour its contents

inside the Cp tubes. Ensure fast filling up of the tubes as the solution keeps flowing out from the bottom. Take the stopwatch and note the time (in seconds) taken by the solution to flow from the top mark to the bottom mark. Similarly note the time taken for the second Cp tube. Repeat the procedure for the other samples. Now using the chart calculate the Cp of the sample.

Using the formula,

DP = (1000 * log Cp) – 400, Degree of polymerization is calculated.

4.7 Sampling plan to carry out the Cp experimentation exercise:

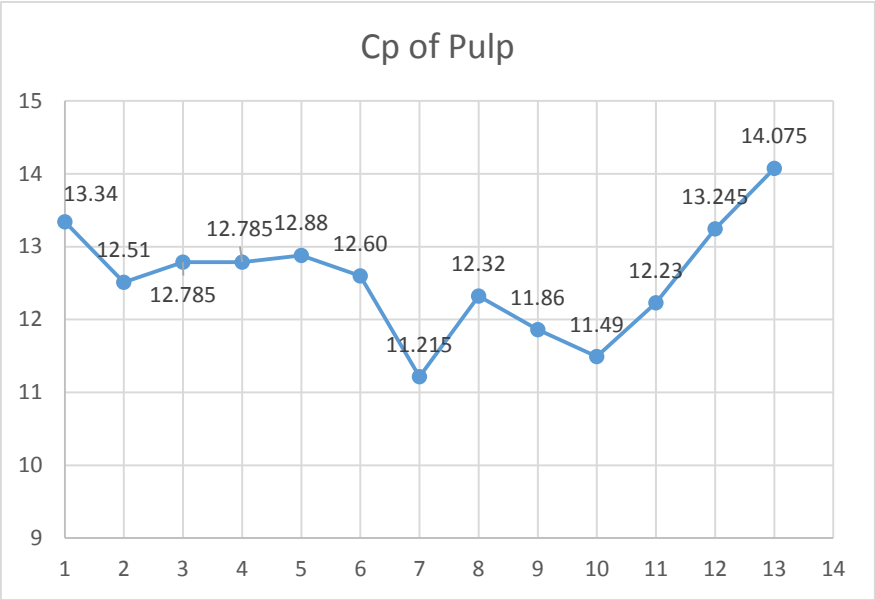
Table 4.3: Sampling plan

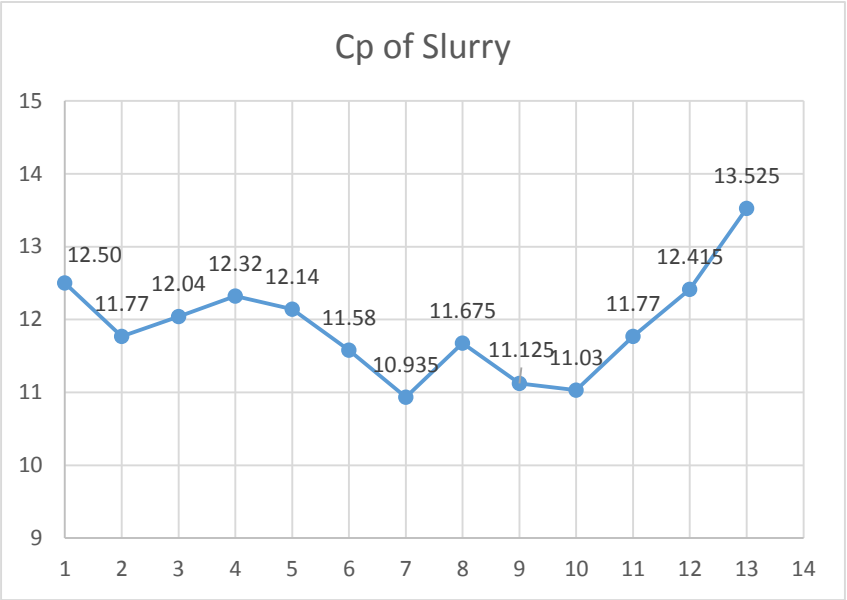
Area	Relevant Time	Time for consideration	Parameters	Collection
Pulp added	0 min	0	Cp of pulp	Collect Pulp sample
NMMO added	0 min	0	NMMO temperature, NMMO conductivity	Record NMMO temperature added
Batch Made	30 min	30 min	Slurry temperature, Cp of slurry, % Cellulose in slurry	Record Slurry temperature, Collect slurry sample
Batch dumped	5 min			
Reach Slurry Feed Pump	60 min	1 hr. 35 min	Slurry Feed Rate, Slurry temp by RTD	Record the said parameters
Reach WFE	6 min			
Reach till cone	8 min	1 hr. 49 min	WFE Rotor RPM, WFE Shell Temperature, WFE Vacuum, Refractive Index	Record the said parameters
Cone to Screw	40 min	2 hr. 29 min	Screw RPM	Record the parameter
Reach Spinning pump	30 min	3 hr.	Cp of dope, % cellulose of dope, MFI of dope	Collection of dope

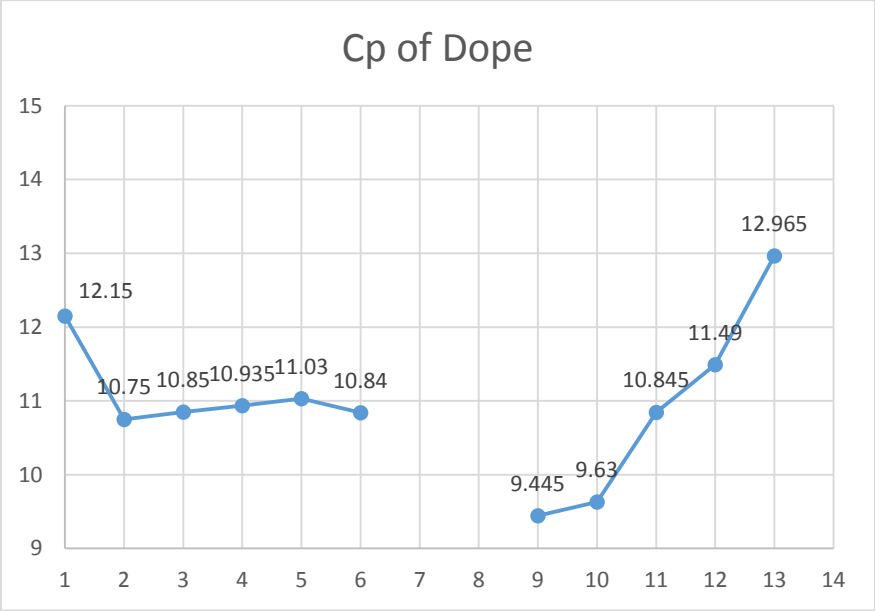
4.8 Results and Analysis of data obtained from Cp experimentation:

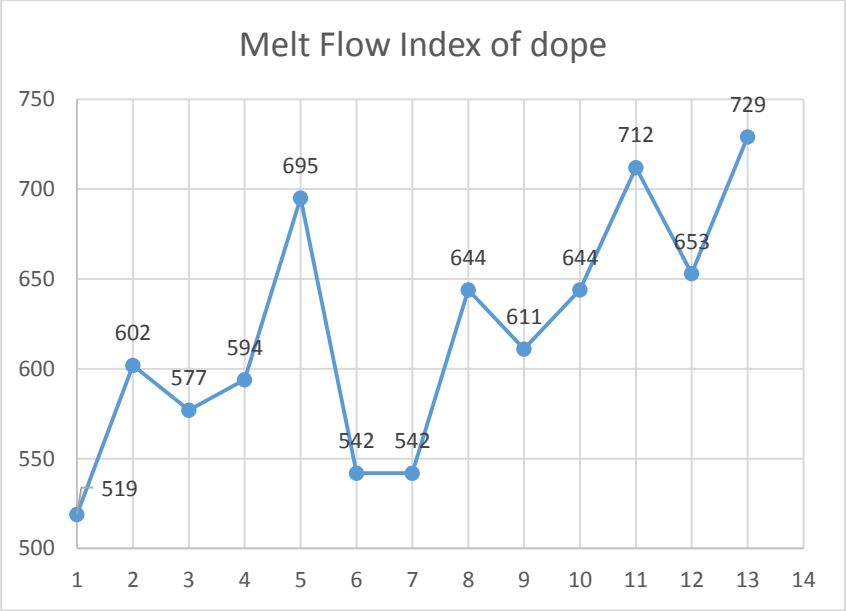
Table 4.4: Experimentation to determine Cp and DP of pulp, slurry, dope

Date	Time for pulper	Cp of pulp	Cp of slurry	Cp of dope	% Reduction of Cp in slurry w.r.t pulp	% Reduction of Cp in dope w.r.t pulp	DP of pulp	DP of slurry	DP of dope	% Reduction of DP in slurry w.r.t pulp	% Reduction of DP in dope w.r.t pulp	% Cellulose in slurry	MFI of dope	% Cellulose in dope
24.11.15	8.00 am (P1)	13.34	12.50	12.15	6.297	8.921	727	697	683	4.127	6.052	9.02%	519	11.02%
25.11.15	8.05 am (P2)	12.51	11.77	10.75	5.915	14.069	697	672	633	3.587	9.182	9.05%	602	11.07%
26.11.15	7.50 am (P2)	12.785	12.04	10.85	5.827	15.135	707	679	621	3.960	12.164	9.09%	577	11.01%
27.11.15	8.05 am (P1)	12.785	12.32	10.935	3.637	14.470	707	690	637	2.405	9.901	8.992%	594	11.01%
28.11.15	8.22 am (P2)	12.88	12.14	11.03	5.745	14.363	711	683	641	3.938	9.845	9.12%	695	11.20%
30.11.15	8.53 am (P1)	12.60	11.58	10.84	8.095	13.968	700	664	633	5.143	9.571	9.08%	542	11.14%
2.12.15	8.45 am (P1)	11.215	10.935	NA	2.497	NA	649	637	NA	1.849	NA	8.78%	542	11.09%
3.12.15	8.50 am (P2)	12.32	11.675	NA	5.235	NA	690	668	NA	3.188	NA	8.992%	644	11.19%
21.12.15	8.35 am (P1)	11.86	11.125	9.445	6.197	20.363	676	645	573	4.586	15.237	9.01%	611	11.26%
22.12.15	8.30 am (P1)	11.49	11.03	9.63	4.003	16.188	661	641	582	3.026	11.952	8.997%	644	11.22%
23.12.15	8.58 am (P1)	12.23	11.77	10.845	3.761	11.325	686	672	633	2.041	7.726	8.76%	712	11.13%
24.12.15	8.50 am (P1)	13.245	12.415	11.49	6.267	13.250	721	693	661	3.883	8.322	9.08%	653	11.10%
25.12.15	8.50 am (P2)	14.075	13.525	12.965	3.908	7.886	749	730	714	2.537	4.673	8.89%	729	10.74%


Cp of Pulp
15
14
13
12
11
10
9
1 2 3 4 5 6 7 8 9 10 11 12 13 14
13.34
12.51
12.785
12.785
12.88
12.60
11.215
12.32
11.86
11.49
12.23
13.245
14.075

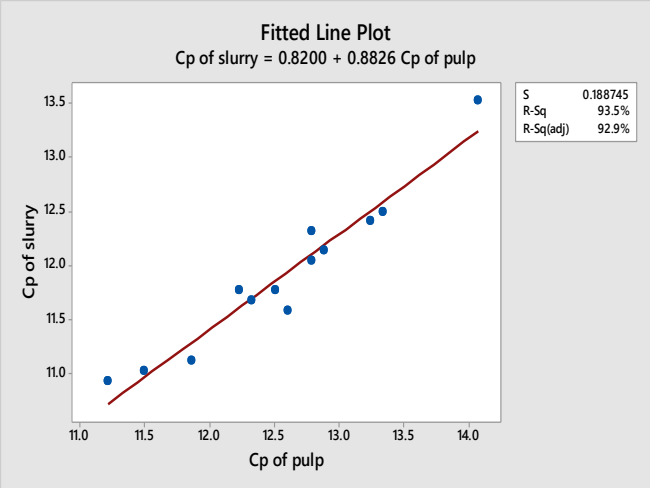

Cp of Slurry
15
14
13
12
11
10
9
1 2 3 4 5 6 7 8 9 10 11 12 13 14
12.50
11.77
12.04
12.32
12.14
11.58
10.935
11.675
11.125
11.03
11.77
12.415
13.525

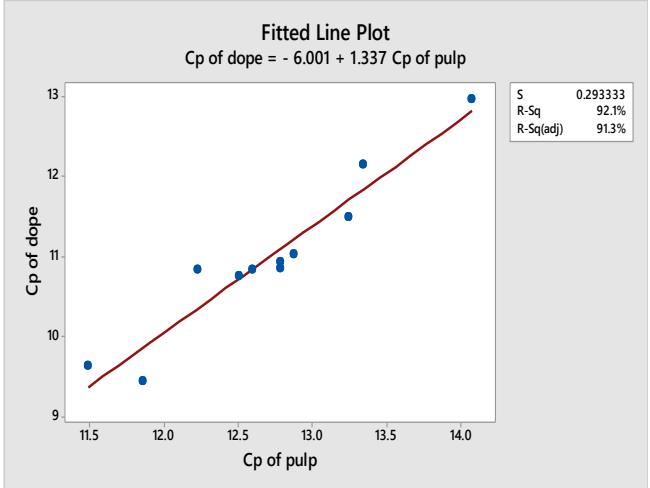

Cp of Dope
15
14
13
12
11
10
9
1 2 3 4 5 6 7 8 9 10 11 12 13 14
12.15
10.75
10.85
10.935
11.03
10.84
9.445
9.63
10.845
11.49
12.965


Melt Flow Index of dope
750
700
650
600
550
500
1 2 3 4 5 6 7 8 9 10 11 12 13 14
519
602
577
594
695
542
542
644
611
644
712
653
729

Analysis of data and plots plotted above-

From the above plots, it can be seen that the trend for Cp of pulp and slurry is almost similar while that for Cp of dope is a bit different. The trend for Cp of dope from **point 10** becomes similar as that of Cp of pulp and slurry i.e. starts increasing. From the plots below it can be understood that Cp of pulp is having a clear effect on Cp of slurry, dope.





The main objective of this work was to observe the effect of Cp of Pulp, slurry and dope on Melt Flow Index (MFI). No continuous trend was observed except for **points 5, 8, 12, 13** where high Cp of pulp gave high values of Melt Flow Index**.** Generally speaking, Cp of pulp has an effect on MFI as was seen some days ago. There was a sudden increase in MFI beyond 1000 on 24-25 December. The reason identified for this abnormal increase after going through several parameters such as Shell Temperature profile, Rotor rpm, Slurry temperature, Cp of pulp used was that rest of the parameters were usual and only change found was Cp of pulp having a higher value around 12.4 (Sappi specification). In order to decrease the MFI the quantity of pulp was reduced. The MFI along with **% Cellulose in Slurry and Dope** got reduced as seen from the above data. Therefore MFI has a relation with Cp but it is not prevalent from the complete data given above. The perfect example for this trend deviation is **point 11 and 12** where lower Cp value gave us higher MFI and vice-versa. It should have been opposite since both Cp and Shell temperature was higher for point 12. But that was not the case. So either there was an error in calculation of MFI or Cp or some other process change

has happened to cause the said shift in trend. Therefore the relation can be seen only from isolated data points.

Even the % Reduction of Cp & DP in Dope w.r.t pulp is not constant and is seen to be varying according to data given above. % Reduction of Cp & DP of slurry was found to be more constant as compared to % Reduction of Cp & DP of Dope with each sample. This evaluation tells us that something in the process is causing uneven degradation of Slurry to dope. That uneven process condition is identified as the **Slurry Temperature** and the reason for stating this can be understood from the following section.

4.9 Characteristics of 'good' dope required for stable spinning:

Melt Flow Index of dope = 525 - 625 (Limits have been changed recently as % Cellulose has increased (On the basis of Lab results)

% Cellulose in dope = 10.8 – 11.2%

RI= 1.4845 – 1.4850

NMMO % in dope = 75.5 – 76%

Precursor of dope:

Slurry temperature = not known

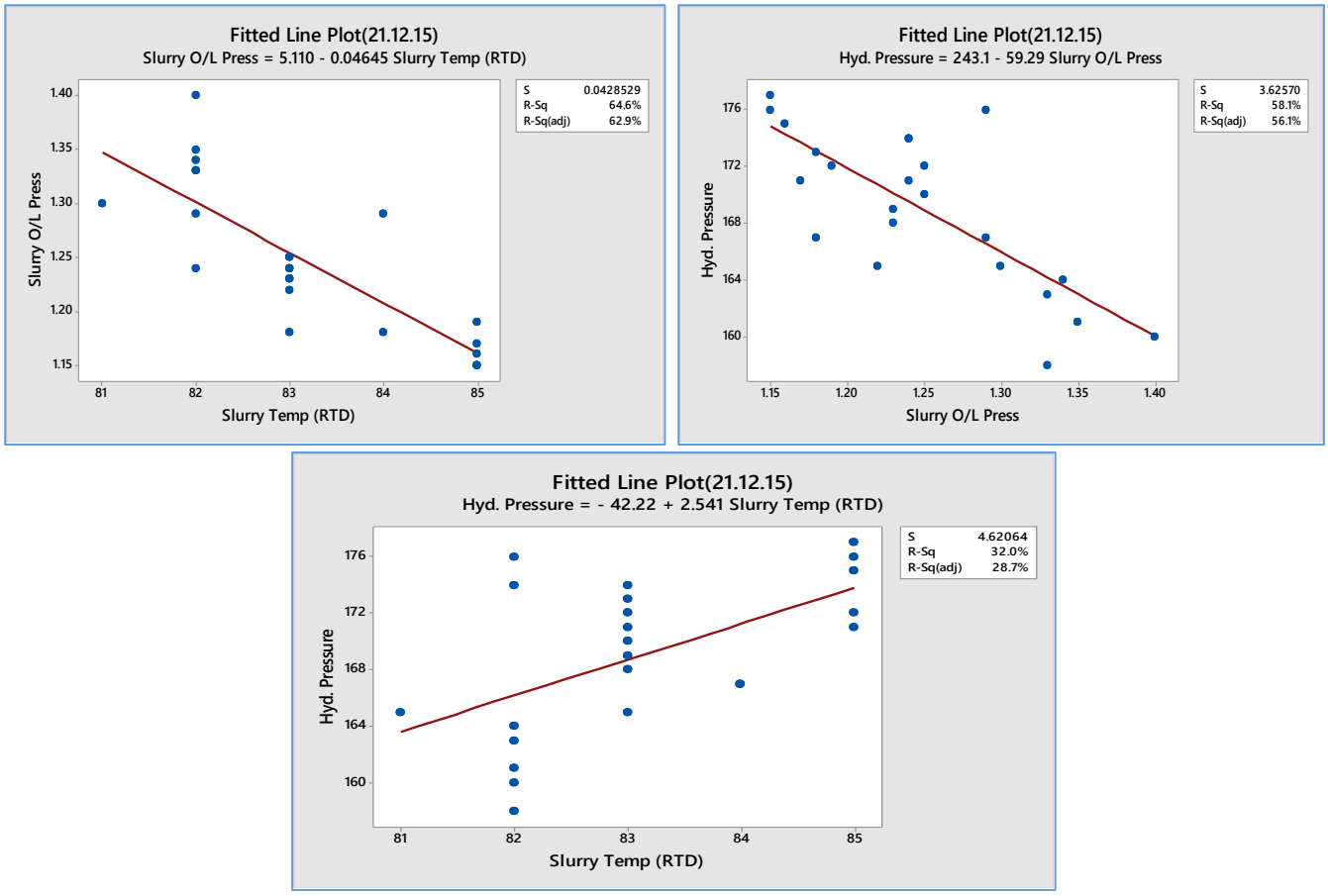
NMMO temperature = not known

Pulp weight= varies with process conditions (Spg. Faults, % cellulose in dope etc.)

Moisture in pulp- 6.5 – 7%

Alpha- cellulose = 92 – 93%

4.10 Effect of Slurry temperature and its maintenance:



Correlation between Slurry Feed Pump O/L Pressure and Slurry Temp-

As evident from the plot above, an increase of Slurry Temperature induces a decrease in Slurry Feed Pump O/L pressure. When the temperature of the slurry increases, its viscosity gets affected and it then becomes easier to pump. This is evident from the drop in pressure and increase in Slurry flow rate. The flow rate increases to around 2.88 - 2.95 m^3/hour (Slurry Temperature= 84 ^{0}C) against a set point of 2.8 m^3/hour. Whenever the slurry temperature is low, pumping becomes difficult which is characterized by Pressure developed by pump. Even the flow rate reduces to 2.52 - 2.60 m^3/hour (Slurry Temperature= 78 ^{0}C).

Correlation between Hydraulic Pressure and Slurry Feed Pump O/L Pressure-

Now as seen from the above plot, an increase in Slurry Feed Pump O/L Pressure brings about a change in the Hydraulic Pressure. From the previous plot it can be seen that one of the reasons

for Slurry Feed Pump O/L pressure to be high is lower Slurry temperature. As already said, if the Slurry Feed Pump O/L pressure increases, the feed flow rate decreases and as a result less amount of slurry reaches the WFE and therefore Hydraulic pressure decreases. Similarly a decrease in Slurry Feed Pump O/L pressure characterizes an increase in flow rate of slurry which then increases the Hydraulic Pressure. The increase in Hydraulic pressure also has another reason. When slurry having a higher temperature reaches WFE, less time is required for the gradient to balance. Thus for the same residence time the dope starts to thicken which also contributes to increase in Hydraulic pressure.

Table 4.5: Comparison of increase/decrease in amount of slurry entering WFE against Set Point due to variation in Slurry temperature

Situation	**Lower than Set Point when Slurry Temperature decreases**	**Set Point**	**Higher than Set Point when Slurry Temperature increases**
Volumetric Flow Rate	2.6 m^3/hour	2.8 m^3/hour	2.95 m^3/hour
Mass Flow Rate	2.6*1.1*1000 kg/hour =2860 kg/hour	2.8 * 1.1*1000 kg/hour = 3080 kg/hour	2.95*1.1*1000 kg/hour = 3245 kg/hour
Difference w.r.t Set Point in kg/hour	=3080-2860 = 220 kg/hour less supply		=3245-3080 = 165 kg/hour more supply
Difference w.r.t Set Point in kg/10 min	=220/6 kg/10min less supply =36.67 kg/10 min less supply		=165/6 kg/10min more supply =27.5 kg/10 min more supply

Thus for decrease/increase in slurry temperature, significant amount of slurry is entering less/more than desired set point. This affects other parameters which will be discussed below.

The variation of slurry temperature causes variations in-

- Slurry Feed pump O/L Pressure
- Slurry Feed flow rate
- Hydraulic Pressure
- Refractive Index
- Shell Temperature
- Melt Flow Index

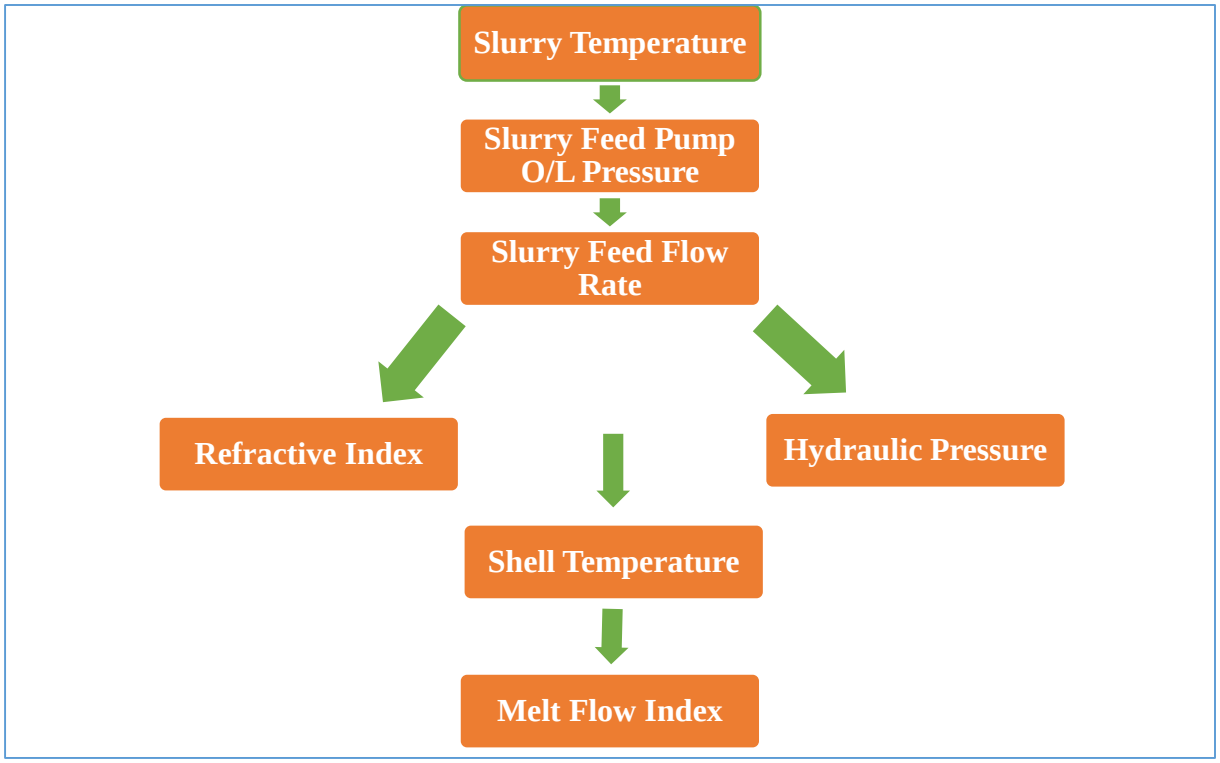


Figure 4.7: Flow Chart sequence of NMMO temperature affecting other parameters

As differential amount of slurry enters the WFE, it affects Hydraulic pressure, Refractive Index etc. As different amount of slurry enters, it causes temperature difference to the existing stock inside the WFE. Due to this there is a change in RI. When there is a variation in RI or Hydraulic Pressure, the Shell temperature is varied to keep it in limits. When Shell temperature is varied, it directly varies the amount of effective evaporation which is responsible for Melt Flow Index.

Now, the slurry temperature in the pulper is directly dependent on NMMO temperature (*see plot below*) and temperature attained during the pulping operation. The temperatures given in the plot below are measured at the pulper. **The NMMO temperature as measured by a mercury thermometer during experimentation was found to be varying between 52 ^{0}C to 67 ^{0}C contrary to the NMMO temperature sent at 74 – 80 ^{0}C from the Feed to Pulper tank.** There can be primarily two reasons namely-

i. The line bringing in NMMO is quite long. Though it is insulated, it is exposed to ambient conditions and approximately 20 ^{0}C is lost during the flow to the overhead tank above the pulper.

ii. The overhead tank above the pulper is not insulated (See image below) and can be insulated by asbestos rope. The sampling was done as soon as NMMO started filling in the tank but due to bulk quantity of NMMO, the temperature loss is also fast.

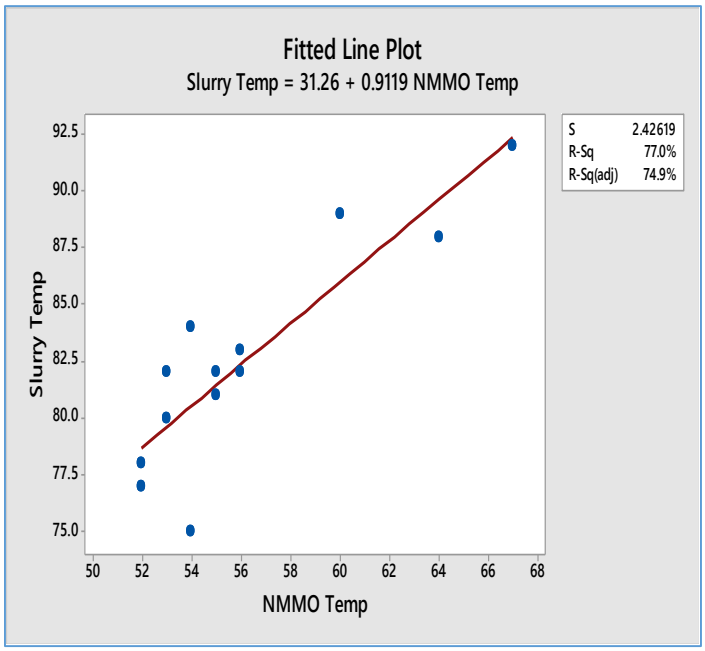


Fig: NMMO temperature vs. Slurry temperature

Fig: NMMO overhead tank in 10 TPD pulper section

In order to mitigate this problem two solutions are proposed in the subsequent pages

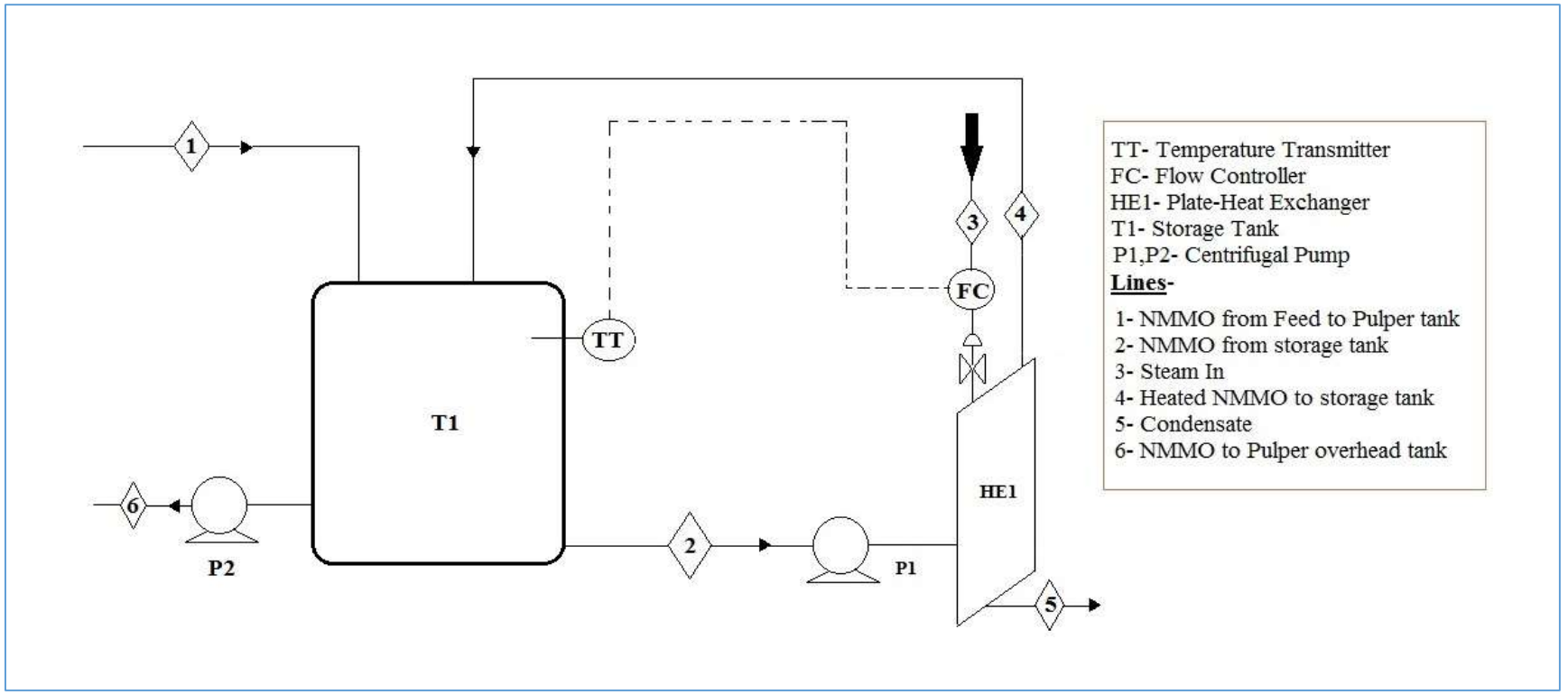


Figure 4.8: Schematics to mitigate variation in NMMO temperature

In the current process, NMMO from the Feed to Pulper tank directly enters the overhead tank above pulper. Instead as shown in the above PFD, a storage tank of about 4 m^3 capacity will have to be built near the Pulper section. The NMMO from Feed to Pulper tank will come in this storage tank. This NMMO will be passed through a PHE employing steam as the utility. The heated NMMO will enter the same tank and the temperature will be monitored by a RTD. This RTD will have a **cascade control** with the valve regulating steam and the temperature of NMMO will be maintained accordingly. The NMMO to the Pulper overhead tank will be sent from this storage tank.

The construction is also easy. Only the line bringing in NMMO has to be bifurcated just before the manual valve which is marked as Point 2.

Point 1: NMMO line from the storage tank will be joined here. A hand operated control valve similar to current operation is to be put here.

Point 3: This is the line from the Feed to Pulper tank. This line instead of directly going into the overhead tank, will go to the NMMO storage tank as shown in the above schematic. Rest of the arrangement will remain absolutely the same.

Figure 4.9: Valve restructure point of 10TPD pulper overhead tank

Another approach can be to install a heating coil inside the NMMO overhead tank along with insulating the tank. A RTD have to be installed to monitor the NMMO temperature.

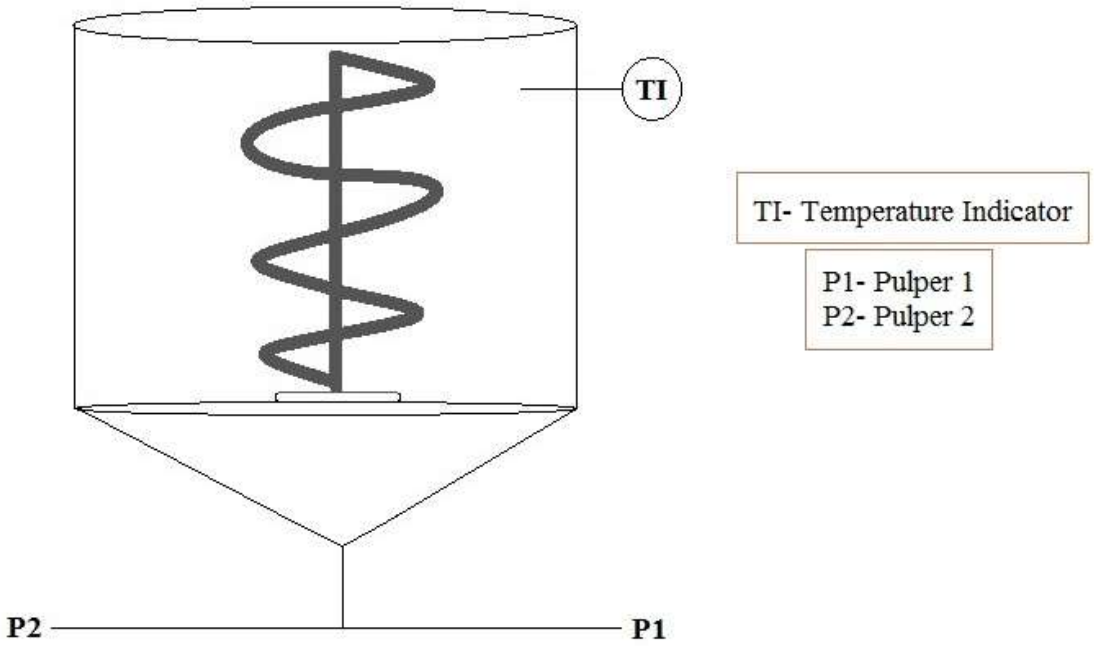


Figure 4.10: Alternate arrangement schematics to maintain NMMO temperature

This type of arrangement can be seen in 0.5 TPD recovery section where the balancing tank has a steam coiled arrangement. The only major constraint with this approach is that sufficient residence time have to be provided after filling the tank for attainment of uniform NMMO temperature

Now after the pulping operation, the slurry is sent to the hopper which is jacketed. But the hopper heating circuit temperature (hot water) varies around - 8 to + 6 ^{0}C from the Set point (Continuous deviation every minute).This was reported and the instrumentation engineer inspected the control valve. There was a problem found with the regulation capability of the control valve. So now this control valve is run in manual mode and the control valve opening % is adjusted by the operator according to Process value of heating circuit of Hopper which needs precision and awareness. Other problem for this variation is the steam trap for PHE 6 (heating circuit of hopper). The steam trap is rusted and backflow of condensate is taking place. As a result of this the bypass valve is opened which leads to loss of steam along with variation in temperature. Also steam keeps leaking from the steam control valve. The images are attached below-

Fig 4.11: Steam trap and bypass valve of PHE 6

Fig 4.12: Steam Control valve of PHE 6

In order to control the hopper temperature, the control loop is arranged according to the RTD placed near the PHE. Instead the control loop can be rearranged according to the RTD placed in the Hopper monitoring the hot water temperature. This can be seen from the schematic below-

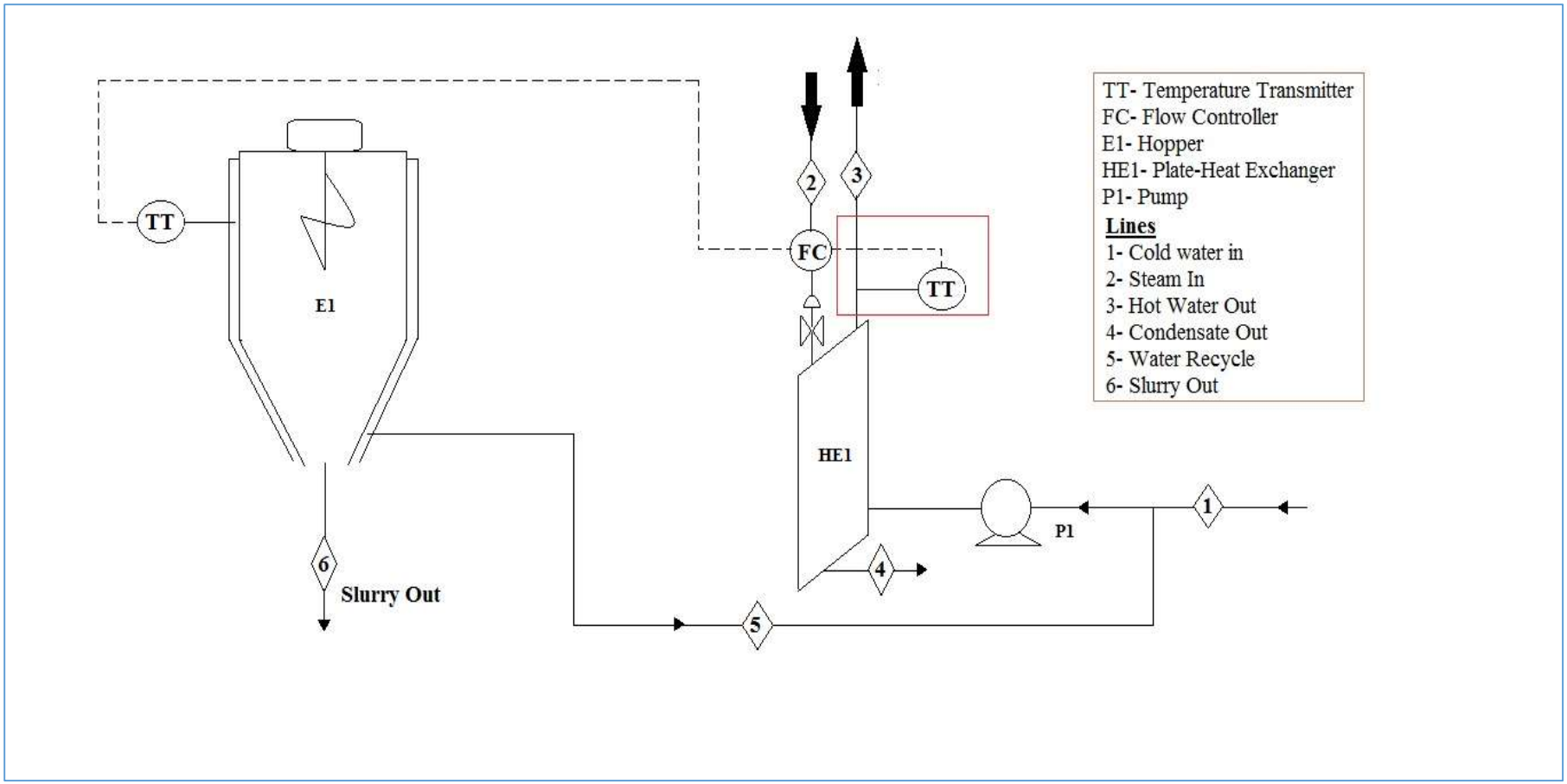


Figure 4.13: Schematics to maintain Hopper temperature

As seen from the above PFD, the area marked in red is the current looping reference. Instead the RTD installed in the hopper section can be used as the looping reference as that RTD will give the exact temperature at the hopper and control of the temperature in the heating circuit can be done accordingly and more accurately.

After the schematic regarding the Hopper temperature is proposed, then the slurry line needs to be jacketed as the importance of the slurry temperature is already discussed above. In order to do this a schematic has been proposed below-

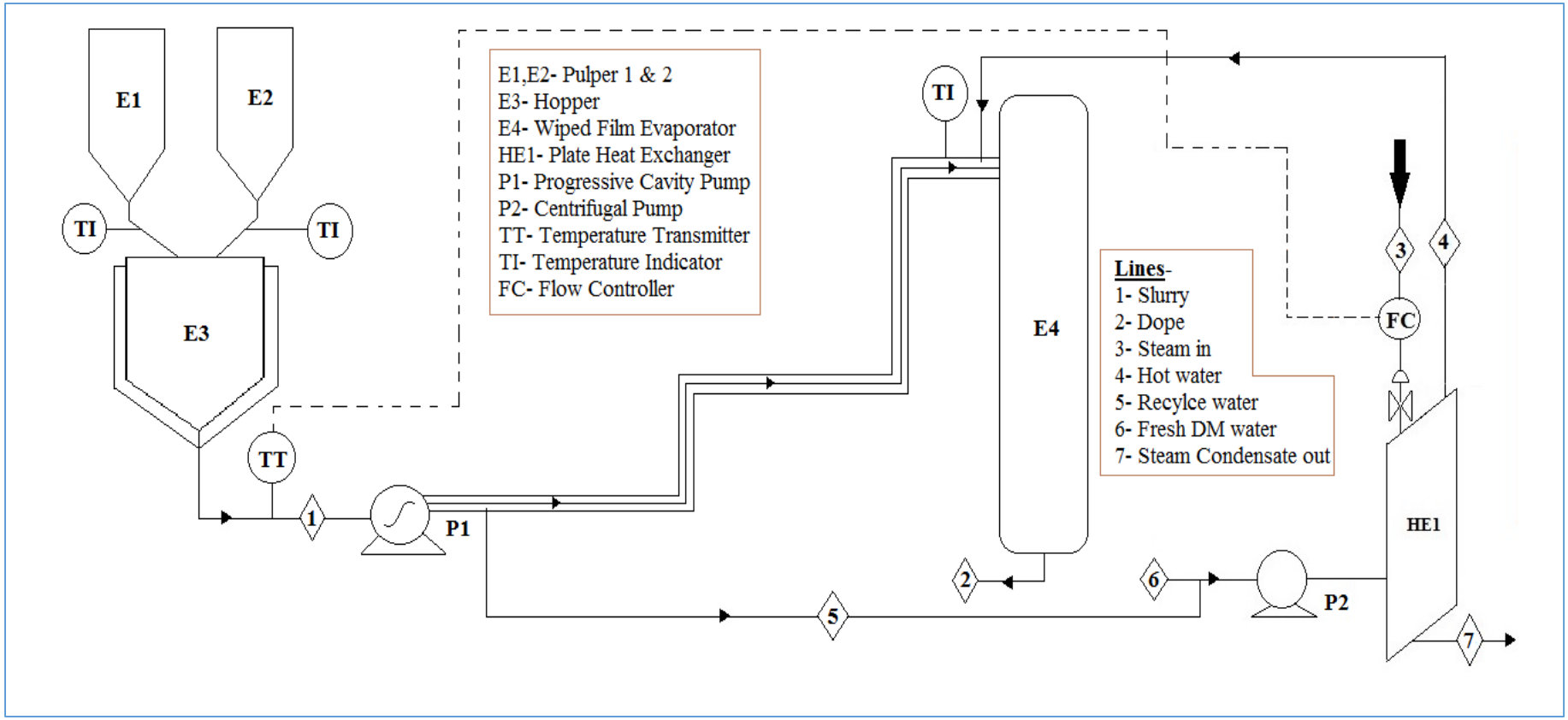


Figure 4.14: Schematics to maintain Slurry temperature

In the above schematic it has been proposed that two RTD's should be installed in the lines coming from the Pulper to Hopper. Then the exact temperature can be monitored. Now the line after the Progressive cavity pump needs to be jacketed as the line has 7 bends which leads to loss in temperature. In order to do this hot water from the PHE is to be sent to the point where slurry enters the

WFE. Due to gravity the water will flow down and then the pump will recycle the water back to the PHE and the flow will be continued. The hot water temperature will be monitored by the slurry outlet temperature i.e. the control loop will be arranged with reference to the RTD placed after the Hopper but before the Progressive cavity pump. This will facilitate proper control of hot water temperature as the point at which RTD is placed will be at constant temperature according to the schematic proposed above. Also a RTD is needed to be put near the slurry inlet point to WFE. This will give us a better understanding as to whether the temperature is being maintained or not.

4.11 Identification of causes for variation of Shell temperature:

Firstly two questions are to be answered after proper investigation-

i. When the Set point (SP) of Shell temperature is varied then how much time does it take to get reflected in the Process Value (PV)?

Answer- Whenever the Set Point (SP) of Shell Temperature is varied it takes around 1.42 - 1.64 minutes to get reflected in the Process Value (PV).

ii. After changing the Set Point (SP) of Shell temperature & Hopper (heating circuit) temperature, how much oscillation of Process value happens around the Set Point?

Answer-

Deviation recorded for a span of 15 minutes

Shell-I = -1.2 ^{0}C to + 1.8 ^{0}C

Shell-II = - 1.7 ^{0}C to + 2.1 ^{0}C

Shell III = - 1.6 ^{0}C to + 1.9 ^{0}C

Shell IV = - 1.2 ^{0}C to + 1.3 ^{0}C

Hopper (heating circuit) = - 8 to + 6 ^{0}C (Continuous deviation every minute)

Some of the identified reasons for these variations –

a) Steam trap of Shell-I & Shell- III has a problem. It causes backflow of condensate/steam passing with condensate which can be seen from the images below. It also leaks steam. Now this backflow of condensate/steam flowing out causes the steam temperature to vary as the rejected condensate unable to be discharged brings the heat value of steam down.

Fig 4.15(a): Steam trap of Shell-I

Fig 4.15 (b): Steam trap of Shell-III

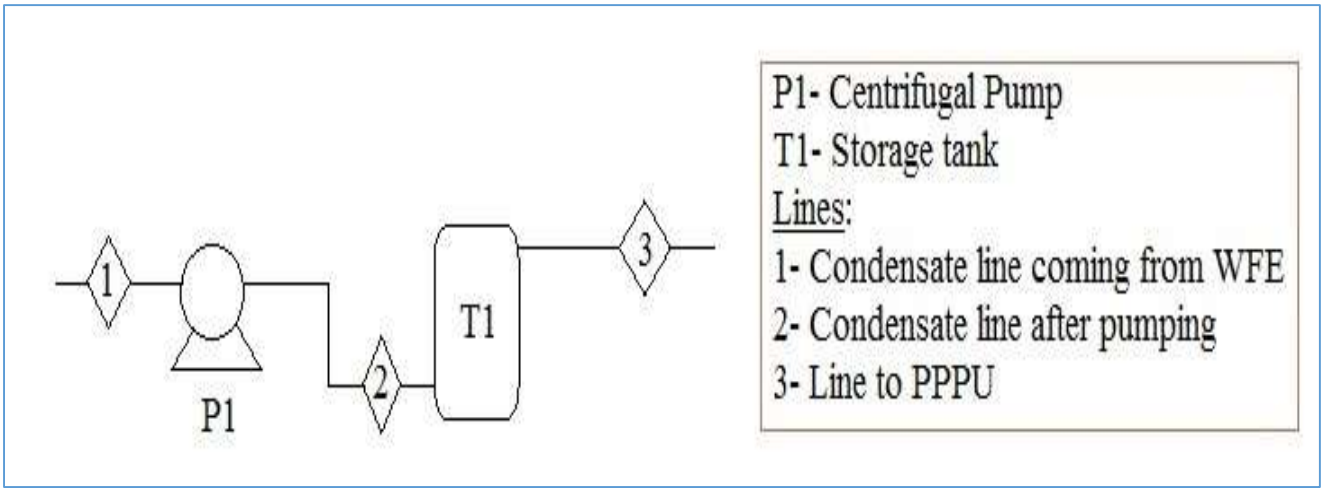
Fig 4.15(c): Opening of both main and bypass valve of steam trap of Shell-III

In order to clear out the condensate which the trap is unable to discharge, the steam trap is run in bypass mode. This when done is observed to increase the other shell's temperature oscillation around the Set Point. When the bypass valve is opened the condensate directly flows out without meeting the steam trap. Since the condensate line is same for all the four this affects other Shells as well.

b) The PPPU (Pressure Powered Pumping Unit) is not able to take in all of the condensate formed because of which the PPPU line is also opened in bypass mode.

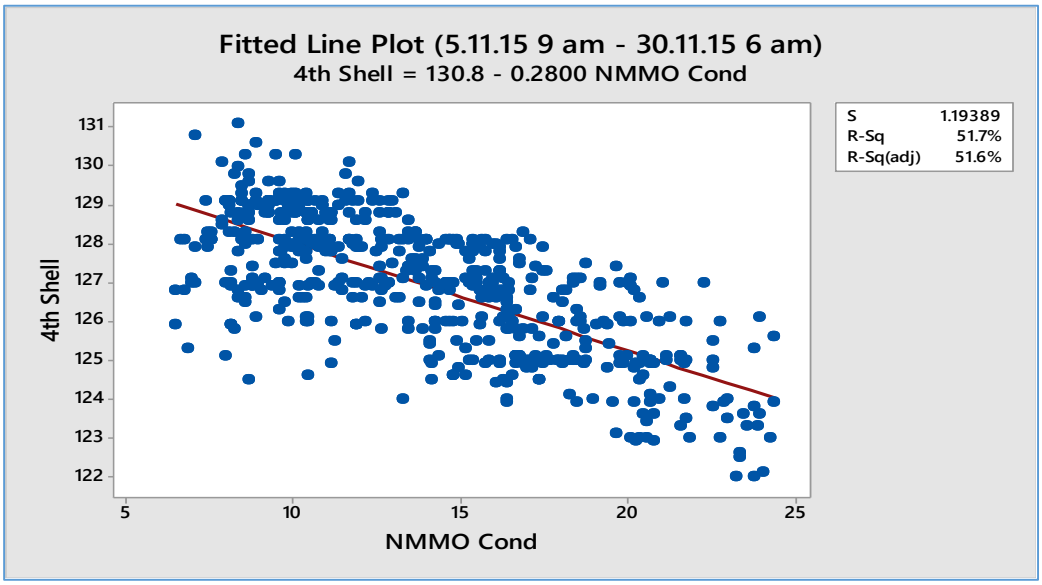
Fig 4.15 (d): Bypass of PPPU opened

PPPU is opened in bypass mode to let out the condensate coming in from the Shells as the unit is not able to suck in all the condensate. If this is not opened then there are higher variations seen in the Shell temperatures. A solution can be to install a small centrifugal pump to suck in all of the condensate and send to a storage tank near the PPPU in the leaching section (*see schematic below*).



As seen from above, the condensate is sucked in by the centrifugal pump and discharged to the storage tank. The PPPU can then take in the condensate from the storage tank.

c)



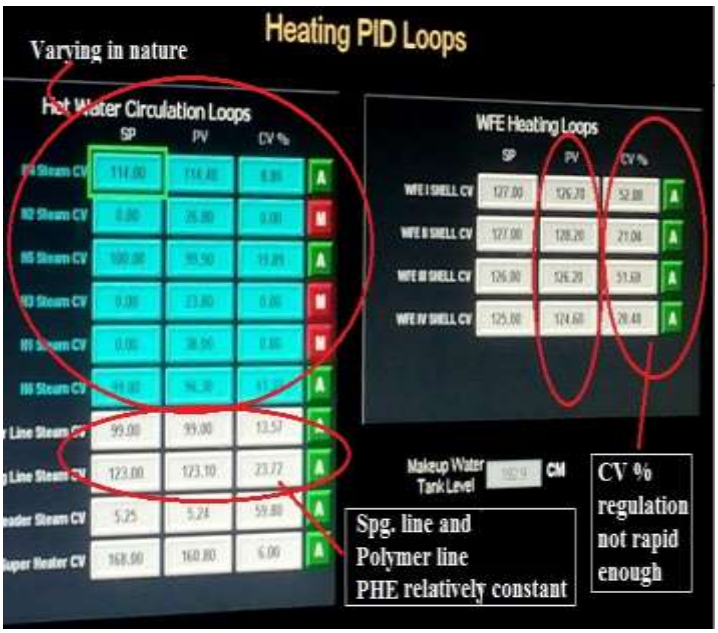
The plot below tells us that when Feed to Pulper NMMO Conductivity increases, the WFE 4th Shell Temperature decreases. This is only possible if RI also increases with conductivity as the operator in WFE section cuts the shell temperature according to an increase in RI i.e. the decrease in WFE 4th Shell Temperature is result of control action taken by the operator. Due to the above situation, the Shell temperature is probably varied.

d) <u>One other observation</u>- Almost all of the heating circuit temperatures after the De-superheater vary. The Spinning line heating circuit and the Polymer line heating circuit

<u>Fig 4.15 (e)</u>: Spg. Line and Polymer Line PHE

<u>Fig 4.5(f)</u>: De-superheater water sprinkling

temperatures relatively do not vary much in comparison to the values of the heating circuit temperatures after De-superheater.



As can be seen from the image, the values prior De-superheater is relatively constant. The reason identified for this variation is that there is a problem with the control valve. The process value as observed has an oscillation of ± 5 ^{0}C over the Set point which is high. By the time the control valve takes action, the value already increases/decreases than the desired value which brings a variation to the heating circuit temperatures post De-superheater.

e) As already pointed above, different Slurry temperatures bring about a variation to the Hydraulic pressure & Refractive Index (RI) and to keep these in limits the Shell temperature is varied.

4.12 Other significant observations & propositions:

a. A pulp shredder can be installed to homogenize the slurry formed. The pulp sheets sent in the pulper after swelling due to NMMO addition is broken down by the action of the agitator. The agitator action does not promise homogenized breaking of the pulp sheets. Therefore a pulp shredder should be installed which will send homogeneous shredded pulp. This in turn will ensure formation of homogeneous slurry.

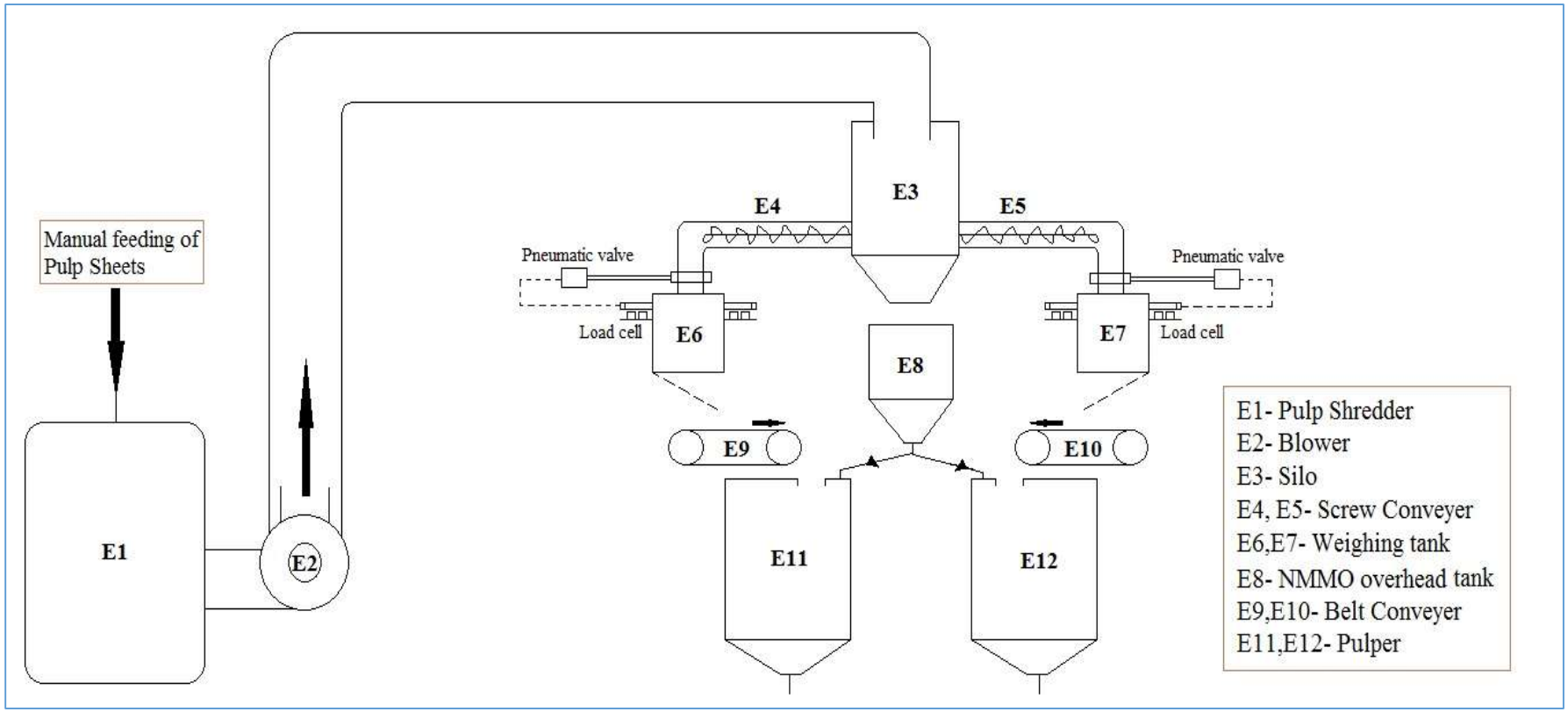


Figure 4.16: Schematics for installation of a pulp shredder

The above image gives us the schematic for installing a Pulp Shredder for preparation of homogeneous slurry. The pulp sheets will have to be manually fed to the pulp shredder. Once shredded, this pulp will be sent forward by a centrifugal blower. The shredded pulp will get collected in a silo. Then the shredded pulp will be sent into a weighing tank connected with Load cells with the help of screw conveyer. This line is regulated by a pneumatic valve which will be actuated by the load cells i.e. when this line is open then the bottom cover of the weighing tank will be closed. The weighing tank is basically hanging in position and connected to Load cells. When the pulp is of the required quantity, then the pneumatic valve closes the line. At this moment the bottom cover opens up and the pulp falls on the belt conveyer which moves the pulp into the Pulper. The NMMO from the overhead tank enters the pulper and batch is prepared.

b. NMMO line from overhead tank to the pulper in the pulper section does not become empty even after complete dumping of NMMO from the tank.

Fig 4.17 (a): View-glass for Pulper-2

Fig 4.17 (b): Solution proposed

This is seen for both the pulper in operation. This may lead to different amounts of NMMO going in for each batch. A solution as seen from the image above can be to tilt the line at an acute angle to drain out complete NMMO. A counter argument can be given that each time the previous batch of NMMO present in the line is pushed out during preparation of fresh batch. But this cannot be validated. Also the NMMO present in the line loses heat. So it is best to modify the design of the line so as to ensure that each time exact 1064 L of NMMO enters the pulper for batch preparation.

c. A meek sound is heard from the rotating area of the Screw Discharge pump (*as seen from image*) in the WFE section. It appears as if air is entering with each rotation of the screw. As the system is under vacuum, this if happening is disturbing the system and allowing vacuum leakage. It can be a reason for the continuous opening/closing of control valve for attaining vacuum.

Figure 4.18: Screw Discharge at the bottom of cone section of WFE

The above reason was identified when there was a drop in vacuum. This was proven as the intensity of the sound along with variation of the control valve % of z- steam ejector decreased after the gland nut was tightened.

d. The fresh Candle Polymer Filter (CPF) ΔP for A side is 34–35 bar while for B side is 20–22 bar. The upper cap for both the CPF is fixed at 60 bar. So when ΔP reaches the upper cap, CPF is changed. That indicates the life of A side CPF is less than B side. The upper cap of both sides should be different i.e. upper cap of A side should be greater than B side.

To exemplify this below data is provided-

5.12.15 2.00 pm B-side

Filter pump O/L pressure = 67.2 bar, delta P = 59.3 bar

After change, Filter pump O/L pressure = 28 bar, delta P = 20 bar

10.12.15 4.00 pm A-side

Filter pump O/L pressure = 67.8 bar, delta P = 59.8 bar

After change, Filter pump O/L pressure = 45.7 bar, delta P = 34.3 bar

e. Slurry enters only through one point into the WFE.

Figure 4.19: Slurry entry points to WFE

Two out of four points were already sealed as slurry was not entering from those points. Now, two points were open (*as seen from image*) through which slurry is believed to enter the WFE. But when the temperature of the inlet points was checked with a Temperature gun it was found that-

Temperature of Entry point 1- 58.6 ^{0}C

Temperature of Entry point 2- 32.3 0C

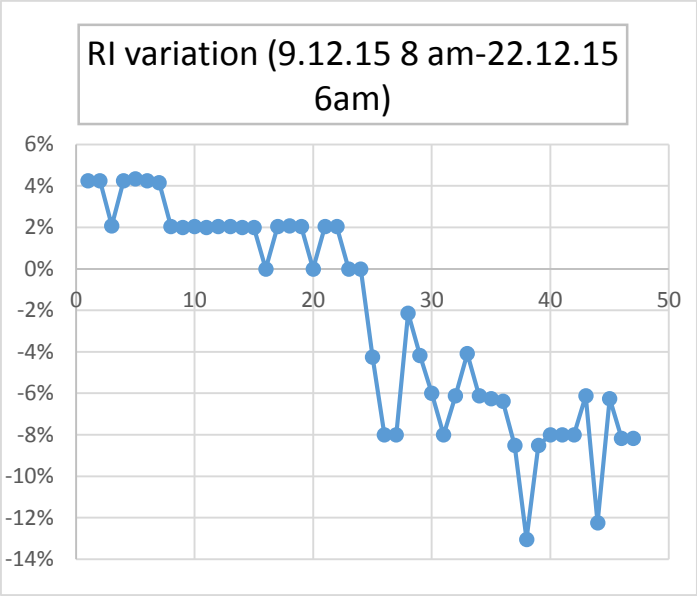
So, it was clear that slurry is entering only through one point.

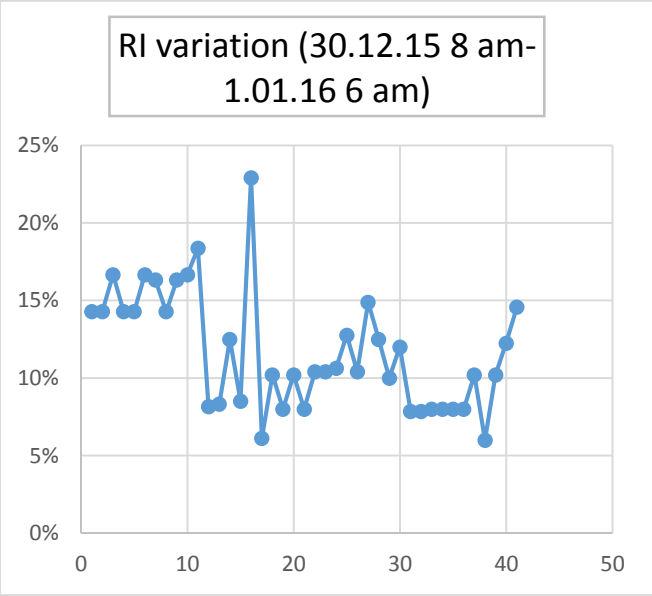
f. Heavy leakage of NMMO observed from the manually operated valve (*as seen from image*) in the pulper section. A cloth was tied to stop the leakage and a bucket was kept to collect the NMMO.

Figure 4.20: NMMO leakage from the hand operated valve

g. Light has been changed according to recommendation which has improved visibility in the pulper region (*see image*).

h. The value of Refractive Index coming in the Historian has a significant difference from the original value displayed in the RI meter and as observed & compared the difference has increased over time (*see plots below*).





4.13 Box-plot validating identified criticalities:

On increase of slurry temperature, the Slurry O/L pressure decreases and simultaneously Slurry Feed flow rate increases. This led to an increase in Hydraulic pressure. The trend is clearly seen from the panel or the data obtained from Historian. But strong Fitted-line plot was not found between slurry temperature and feed flow rate. The reason for this was that the value of feed flow rate was observed to be highly pulsating and due to this for a common value of temperature, values of flow rate was getting repeated. So in order to statistically establish the existing relation, the shifting of mean is shown below with the aid of box plots. When there is an increase in temperature, rather than showing relation with individual values the mean of the values for each temperature is considered thus giving us an outlook of the process.

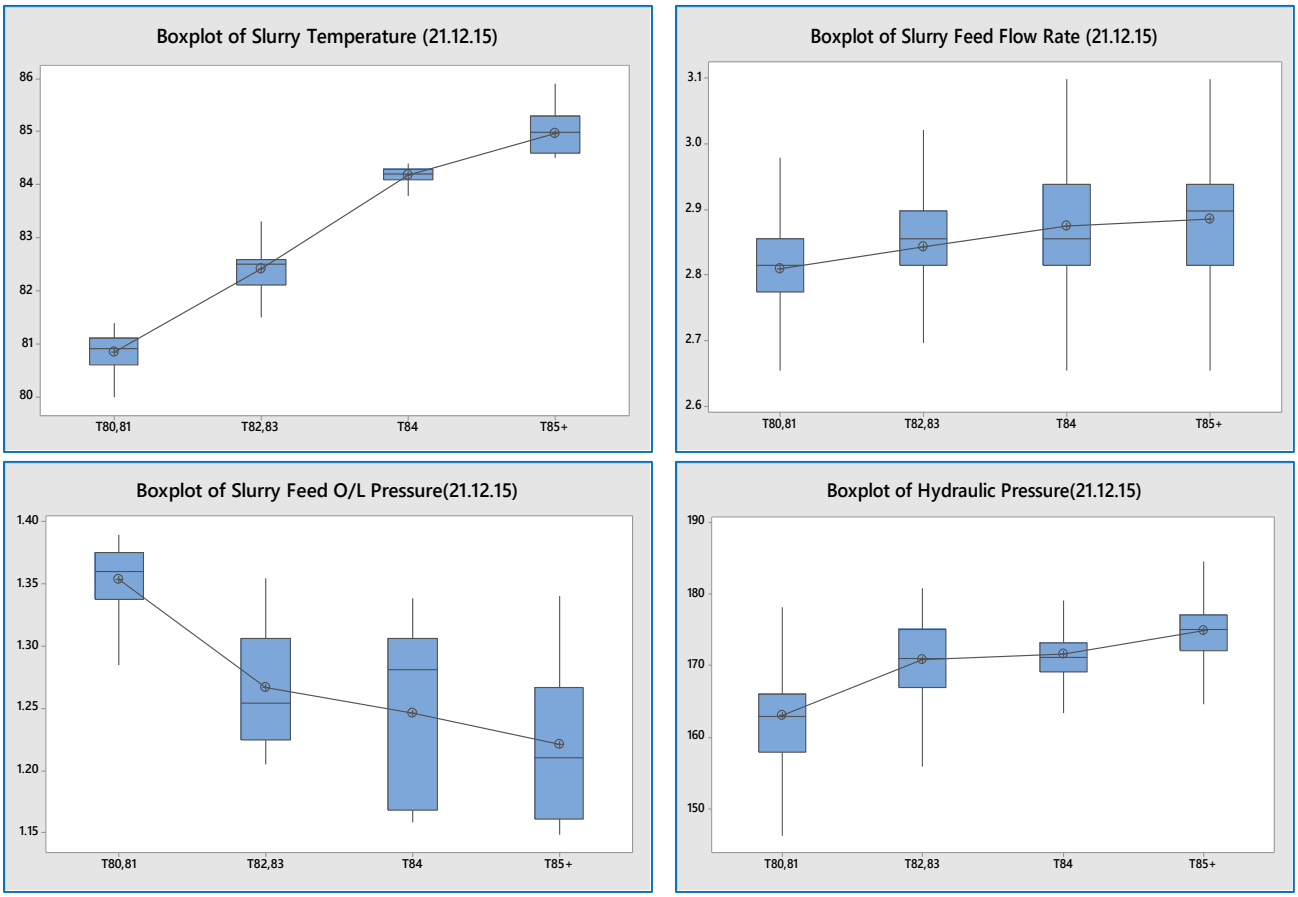


Figure 4.21: Box Plot of different parameters taken at temperatures (80 ^{0}C, 81 ^{0}C; 82^{0}C, 83 ^{0}C'; 84 ^{0}C; 85+ ^{0}C)

The Box Plots above have been prepared taking the respective parameters viz. Slurry temperature, Slurry Feed flow rate, Slurry O/L pressure, Hydraulic pressure. The parameters aforementioned have

been segregated on the basis of temperature rise of slurry ('80, 81 ^{0}C'; '82, 83 ^{0}C'; 84 ^{0}C; 85+ ^{0}C) where '80, 81' ^{0}C & '82, 83' ^{0}C have been clubbed together.

From the box plots above it can be clearly seen that there is a shift in mean i.e. the trend as seen from the panel. As slurry temperature keeps on increasing, the slurry flow rate increases which is a characteristic of decrease in O/L pressure. Thus because of increase in flow, the Hydraulic pressure rises.

Slurry flow rate has an impact on Refractive Index which in turn encourages a change in WFE Shell temperature, the below trend for 21.12.15 has been plotted.

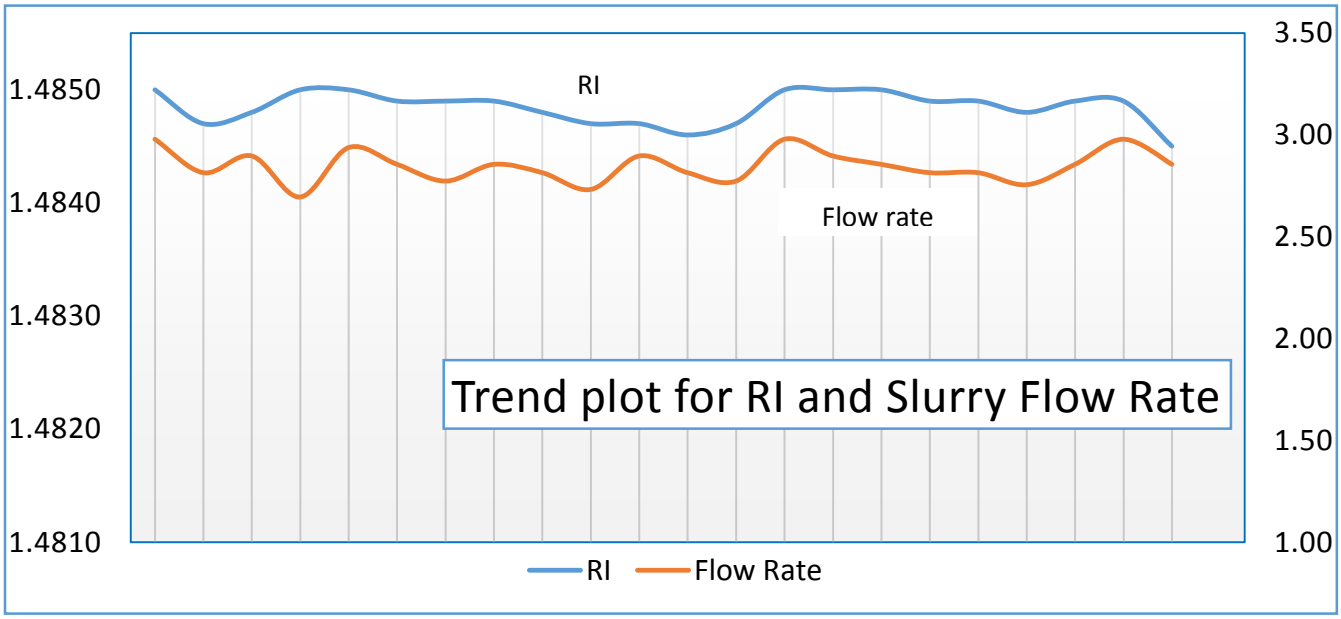


Figure 4.22: Trend plot for RI and Slurry Flow rate

The trend as seen from above tells us that a change in flow rate has an effect on Refractive Index. This is because flow rate is actuated by slurry temperature and a variation in this affects RI.

4.14 Material balance for quantifying effect of slurry flow rate on % cellulose in dope:

The below mass balance has been done in order to quantify the effect of variable flow rate on % cellulose in dope

Basis: Constant % NMMO in dope, Constant evaporation of water

Firstly, % cellulose in slurry was calculated,

% Cellulose in slurry = (Pulp Quantity - Pulp moisture in kg)/ ((NMMO in L * sp. Gravity of NMMO) + (Pulp taken * Moisture % in pulp))

= (116 – (116*0.07))/ ((1064 * 1.1472) + (116 * 0.07)) = 0.0878 = 8.78%

Stream information		Components information			Evaporator	Stream information		Components information		
Name	Mass flow rate,kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr		Name	Mass flow rate,kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr
Slurry	3080	**Cellulose**	0.0878	270.42		H_2O Evaporated		**H_2O**	1	729.47
		NMMO	0.58	1786.4		DOPE	2350.53	**NMMO**	0.76	1786.4
		H_2O	0.332	1023.185				**Cellulose**	0.115	270.42
								H_2O	0.125	293.71

Case-I- Flow rate = 2.8 m^3/hour

Therefore % Cellulose in dope= 11.5 %

Case-II- Flow rate= 2.65 m^3/hour

Stream information		Components information			Evaporator	Stream information		Components information		
Name	Mass flow rate, kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr		Name	Mass flow rate, kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr
Slurry	2915	**Cellulose**	0.0878	255.9288		H_2O Evaporated		**H_2O**	1	729.47
		NMMO	0.58	1690.7						
		H_2O	0.332	968.3712		DOPE	2224.61	**NMMO**	0.76	1690.7
								Cellulose	0.133	295.01
								H_2O	0.107	238.90

Therefore % Cellulose in dope= 13.3 %

Case-III- Flow rate= 2.94 m^3/hour

Stream information		Components information			Evaporator	Stream information		Components information		
Name	Mass flow rate,kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr		Name	Mass flow rate, kg/hr	Name of component	Mass fraction	Mass flow rate, kg/hr
Slurry	3234	**Cellulose**	0.0878	283.9361		H_2O Evaporated		**H_2O**	1	729.474
		NMMO	0.58	1875.72						
		H_2O	0.332	1074.344		DOPE	2468.05	**NMMO**	0.76	1875.72
								Cellulose	0.1003	247.46
								H_2O	0.1397	344.87

Therefore % Cellulose in dope= 10.03 %

Although the above balance considers constant evaporation which is too enthusiastic as WFE Shell temperature is controlled, still it gives us a fair idea that a variation in flow rate indeed has an effect on % cellulose in dope/ % moisture present in dope.

In order to stabilize the flow rate, the oscillation in the control valve opening of stem ejector needs to be stabilized. Since generation of non-condensable gases is not in our control, therefore leakage primarily by screw gland must be taken care of. If this and slurry temperature is maintained, then flow rate will be relatively constant.

4.15 Investigation of presence of a relation between NMMO conductivity & RI:

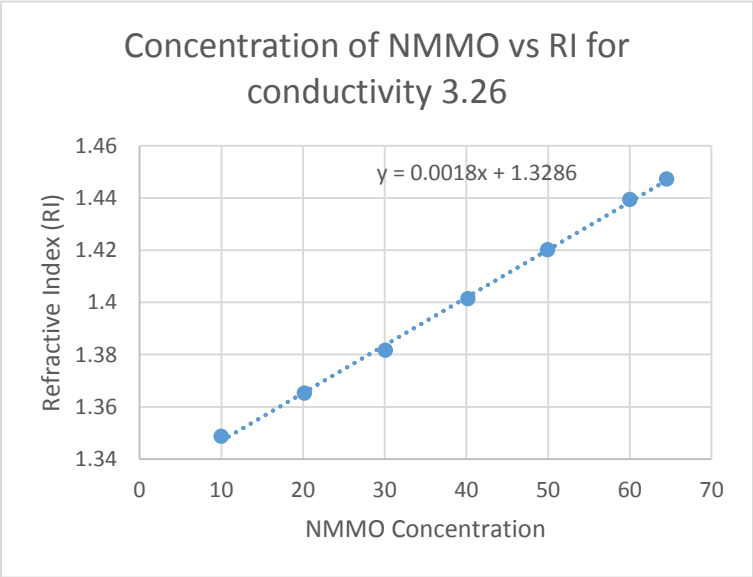
There was a trend seen in the plant that on an increase of Feed ion exchange O/L conductivity there was an increase in Melt Flow Index of dope. Now MFI generally varies if Shell temperature and slurry temperature are varied. It was believed that a variation in inlet conductivity led to a variation in RI. To control RI the shell temperature was varied which leads to a variation in MFI. In order to validate this, an experiment was performed where for a given conductivity (both low and high), data for RI at different concentrations was recorded and plotted.

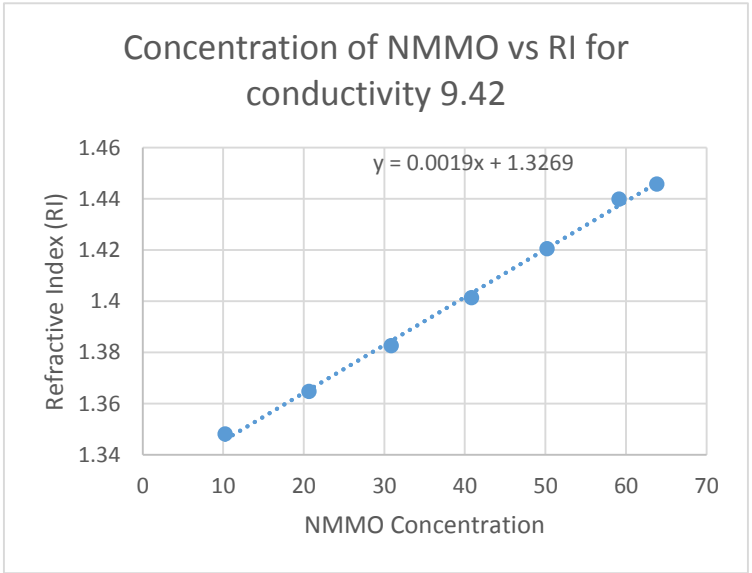
Table 4.6 (a): Data to deduce relation between NMMO concentration & RI for **conductivity 3.26**

NMMO (Theoretical)	Water (Theoretical)	NMMO (Practical)	Water (Practical)	Total (Practical)	Total (Theoretical)	Conc. (Theoretical)	Conc. Practical	RI
Initial	Initial	Initial	Initial	Initial	Initial	64.50%	64.5	1.4458
23.25581395	1.744186047	23.2676	1.7406	25.0082	25	60%	60.01	1.4394
19.37984496	5.620155039	19.3819	5.6614	25.0433	25	50%	49.92	1.4201
15.50387597	9.496124031	15.5121	9.491	25.0031	25	40%	40.18	1.4014
11.62790698	13.37209302	11.6215	13.3029	24.9244	25	30%	30.08	1.3817
7.751937984	17.24806202	7.7572	17.2572	25.0144	25	20%	20.16	1.3652
3.875968992	21.12403101	3.874	21.2194	25.0934	25	10%	9.96	1.3487

Table 4.6 (b): Data to deduce relation between NMMO concentration & RI for **conductivity 9.42**

NMMO (Theoretical)	Water (Theoretical)	NMMO (Practical)	Water (Practical)	Total (Practical)	Total (Theoretical)	Conc. (Theoretical)	Conc. Practical	RI
Initial	Initial	Initial	Initial	Initial	Initial	63.8%	63.8	1.4458
23.51097179	1.489028213	23.5148	1.5233	25.0381	25	60%	59.12	1.4399
19.59247649	5.407523511	19.5991	5.4594	25.0585	25	50%	50.20	1.4205
15.67398119	9.326018809	15.6677	9.3666	25.0343	25	40%	40.80	1.4014
11.75548589	13.24451411	11.7519	13.3421	25.094	25	30%	30.86	1.3827
7.836990596	17.1630094	7.8308	17.2081	25.0389	25	20%	20.62	1.3648
3.918495298	21.0815047	3.899	21.1598	25.0588	25	10%	10.24	1.3482





It was observed that a change in conductivity did not affect Refractive Index (RI). Both RI and conductivity are a function of temperature but they according to the above data are independent of each other.

4.16 Maintenance of conductivity by using different resins in Skid Mounted Unit:

Since there was no concrete explanation available as to why the conductivity was varying Melt Flow Index, therefore it was decided that different resin has to be studied to lower down the ion exchange O/L conductivity. In order to facilitate this study, the skid mounted unit was commissioned. The skid mounted unit is nothing but an ion exchange unit where the feed is spin bath solution having concentration of NMMO around 21 %. The ion exchange unit removes the foreign ions primarily coming from the pulp which are responsible for increase in conductivity of NMMO solution. Pure NMMO has a conductivity of around 5 µs/cm. This conductivity on dilution with water and then run in the system has an increase to around 140 µs/cm. The NMMO solution is then to be deionized for removal of these ions.

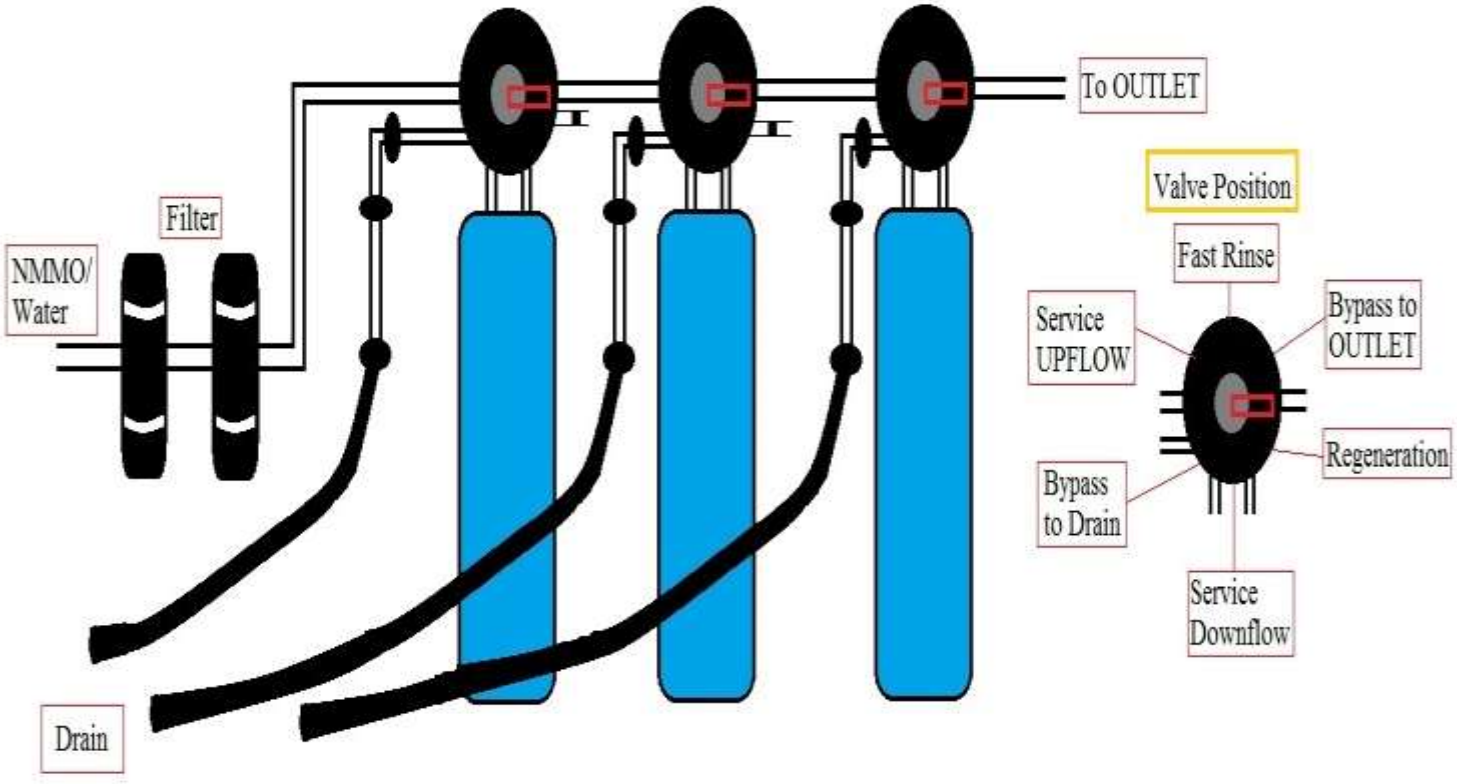


Figure 4.23: Skid Mounted Unit

Running FEED (NMMO (Spin bath solution))-

Before running NMMO solution, the following things must be taken care of-

- Blank is put in the water line
- Blank in the inlet line of NMMO is removed and valve is opened
- Blank in the outlet line is removed and the valve is opened.

Now, the NMMO inlet valve near the skid mounted unit is opened. Also, the inlet/outlet valve near the filter and online conductivity meter are opened. The valve position for the mounted cylinders having resin are then checked. For NMMO feed the valve position should be-

Table 4.7: Valve position for NMMO feed inlet to Skid Mounted Unit

Cylinder 1	Cylinder 2	Cylinder 3
Service Downflow	Service Downflow	Bypass to Outlet

Now the valve at the base of the rotameter is then opened and regulated such that the flow of feed is 500 L/hour. A reading of NMMO inlet and outlet conductivity is recorded each hour and specifically at 10 Bed Volume of feed that is after 1.5 hours of starting the process.

Now, the process is shut down when the outlet conductivity reaches 35 µs/cm (Lab)/ 50-52 µs/cm (online). After this-

- The inlet valve is closed and Blank at the inlet line is put
- Blank at the water line is removed and the valve is opened

Now, water is run in the system (Valve position remains the same) till the point the concentration of NMMO comes down to exact 0.2 % which is seen to happen when the outlet conductivity reaches around 11-12 µs/cm.

Regeneration of the resin in the system-

Now the resin is to be regenerated. During regeneration acid and caustic wash are performed. The acid and caustic are sent inside the cylinders by an educator/ejector system. Water is run at a high flow rate which acts as the motive fluid and then pulls the acid along with it. The acid and caustic

used are kept at around 21 % which when reaches and mixes with water while going inside the cylinder becomes around 5%

Before regeneration-

- The valve must be closed and Blank must be put in the outlet line

Now the regeneration follows fourteen steps which are tabulated below-

Table 4.8: Quantity of chemicals used in regeneration

Regeneration of Anion							
Sample No.	Reagent	Acid (HCl)	Caustic (NaOH)	Water	Total	Flow Rate	Time
1	Water wash (Backwash)	0	0	31	31	500	3 min 45 sec
2	Acid wash I	20	0	63	83	800	4 min 42 sec
	10 minutes hold						
3	Acid wash II	20	0	63	83	800	4 min 42 sec
4	Water Wash I (After acid wash)	0	0	250	250	700	21 min 25 sec
5	Water Wash II (After acid wash)	0	0	83	83	700	7 min 6 sec
6	Caustic wash I	0	20	63	83	800	4 min 42 sec
	15 minutes hold						
7	Caustic wash II	0	20	63	83	800	4 min 42 sec
8	Water wash (After caustic wash)	0	0	250	250	700	21 min 25 sec

Regeneration of Cation							
Sr. No.	Reagent	Acid (HCl)	Caustic (NaOH)	Water	Total	Flow Rate	Time
9	Water wash (Backwash)	0	0	41	41	500	4 min 55 sec
10	Acid wash I	26	0	84	110	800	6 min 18 sec
	12 minutes hold						
11	Acid wash II	26	0	84	110	800	6 min 18 sec
12	Water wash (After acid wash)	0	0	331	331	700	28 min 22 sec
Combined Washing							
Sr. No.	Reagent	Acid (HCl)	Caustic (NaOH)	Water	Total	Flow Rate	Time
13	Anion & Cation column forward washing I	0	0	71	71	700	6 min
14	Anion & Cation column forward washing II	0	0	213	213	700	18 min 15 sec

The valve position to maintained during regeneration are tabulated below-

Table 4.9: Valve position during regeneration

Indion resin			
	Cation	**Anion**	**Blank**
	Valve 1	**Valve 2**	**Valve 3**
Regeneration of Cation			
Water wash (Backflow)	Service upflow	Service to Drain	Service to Drain
Acid Wash	Regeneration	Service to Drain	Service to Drain
Water Wash	Regeneration	Service to Drain	Service to Drain
Regeneration of Anion			
Water wash (Backflow)	Bypass to Outlet	Service upflow	Service to Drain
Acid Wash	Bypass to Outlet	Regeneration	Service to Drain
Water Wash	Bypass to Outlet	Regeneration	Service to Drain
Caustic Wash	Bypass to Outlet	Regeneration	Service to Drain
Water Wash	Bypass to Outlet	Regeneration	Service to Drain
Combined Water Wash	Service Downflow	Service Downflow	Bypass to Drain
Till conductivity 10 µs/cm			

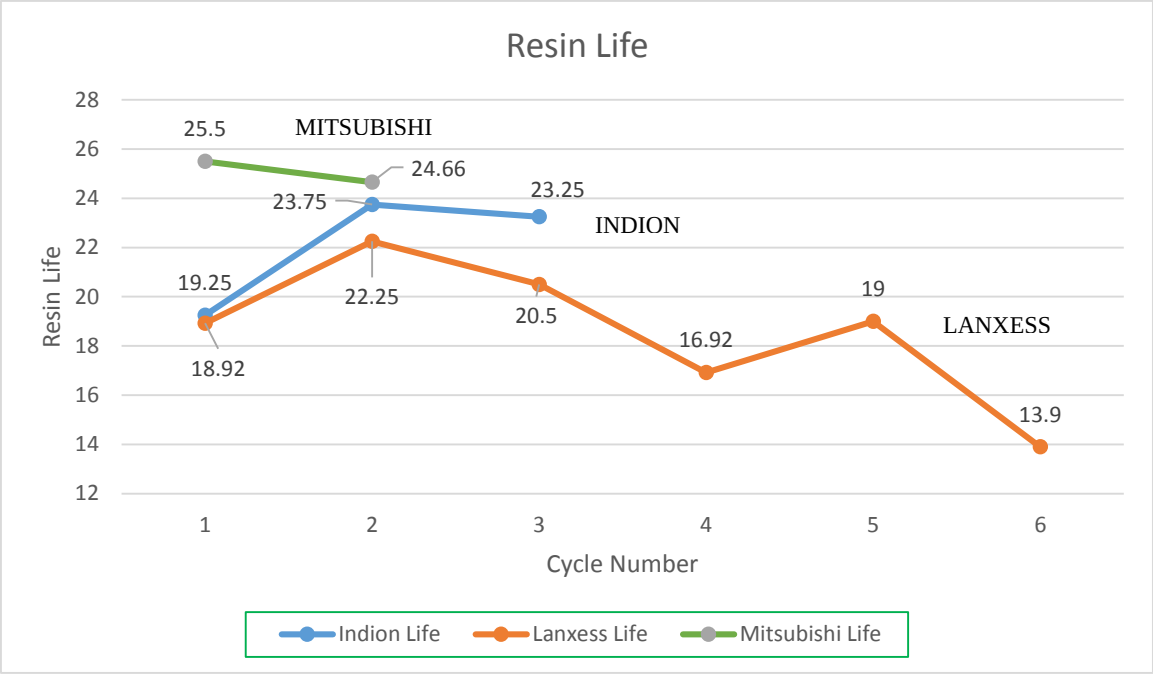
Once the resin is regenerated the cycle is repeated again.

Results from the Skid Mounted system:

There were primarily three resins used for study-

- Lanxess
- Mitsubishi
- Indion

The highest cycle time and best possible removal of ions was obtained using Mitsubishi resin (*see plot below*).



CONCLUSION AND RECOMMENDATIONS

The objective of this project was to reduce the perturbations of the critical parameters of dope preparation section affecting the quality and quantity of Lyocell fiber produced in both the 0.5 TPD and 10 TPD production sections. In order to understand the existing system Process Flow diagrams were drawn which helped in apprehending the current flow scheme. A sampling plan was formulated based on the Fish-Bone diagram conceptualized to identify the critical parameters and eliminate the non-critical ones where Melt Flow Index was selected as the basis for the study as this is the critical to quality parameter of dope. The parameters critical to the process were identified. Post identification data analysis was performed to validate the observations. The perturbations were quantified and solutions to mitigate those have been proposed. In both the 0.5 TPD and 10 TPD section, NMMO and Slurry temperature were found to be the major derailers affecting the succeeding critical process parameters which in turn affected the Melt Flow Index. In order to moderate these derailers, recommendations along with schematics have also been provided. Issues related to conductivity of NMMO was also studied and relevant observations were recorded. In order to decrease the Ion exchange O/L conductivity, different resins were also studied and results pertaining to resin life was also given.

Recommendations:

A: To maintain NMMO temperature, a storage tank has to be built in the pulper section where NMMO will be circulated (*Refer Pg. 56*).

B: To maintain Hopper temperature, the current RTD scheme needs to be replaced with the RTD placed at the hopper (*Refer Pg. 60*).

C: To maintain Slurry temperature, the slurry line needs to be jacketed (*Refer Pg. 61*).

D: In order to homogenize slurry, schematics for installing a pulp shredder has been provided (*Refer Pg. 67*).

E: In order to maintain Shell temperature, worn out steam traps & De-superheater pump are to be repaired to avoid bypass and have a better control.

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