OPTIMIZATION OF VISCOSE PROCESS TO IMPROVE FIBER PROPERTIES

A report

Submitted by:

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CERTIFICATE

This is to certify that the thesis titled **OPTIMIZATION OF THE VISCOSE PROCESS TO IMPROVE FIBRE PROPERTIES** submitted by **KUSHAGRA AGRAWAL** to the University of Petroleum & Energy Studies, for the award of the degree of **MASTER OF TECHNOLOGY** in Chemical Engineering with Specialization in Process Design Engineering is a bonafide record of project work carried out by him under our supervision.

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NOMENCLATURE

acid	Sulphuric Acid
zinc	Zinc Sulphate
salt	Sodium Sulphate
kW	Filter clogging coefficient
W_i	weight of dope collected in T_i time
W'_i	W_i X Correction factor
ΔP	pressure drop
f_d	Darcy's friction factor
L	length of the tube
D	diameter of the tube
P_h	power of pump
ρ	Density of water
V	velocity of water inside pipe
$\dot{q}$	volumetric flow rate
Q	heat flux
m	mass flow rate
c_p	specific heat capacity
ΔT	change in temperature
Δt	change in time
U	Overall heat transfer coefficient
A	area of pipe

ABSTRACT

Viscose process has been widely used in the world to synthesize viscose rayon from the cellulose present in the pulp. The optimization of this process has been of keen interest to the textile industry in order to maximize the profit. The purpose of this thesis is to determine the best process parameters and conditions to optimize the process and identify maximum possible wet strength of the fibre. The thesis can be split in to two major objectives: (a) Method development for determining filterability constant using small Kw (100 g) and (b) optimizing the spinning parameters such as spin bath composition, temperature, etc.

Filterability is one such important parameter which is measured in terms of kW value at a big setup. In order to avoid wastage of prepared dope, a small kW (100 gm capacity) equipment was standardized w.r.t. the previously existing 2 kg set up. It was observed that despite the use of a correction factor, the flowrate of the two setup were not matching. The flowrate of the small setup was decreasing faster than the flowrate of big setup. However, this difference of flowrate did not prove to be an obstacle towards the standardization.

As a part of spinning parameter optimization, several trials were conducted with varying spinbath composition. These trials had to be conducted at a constant temperature, by means of an indigenously designed heat exchanger in the spin bath trough. Based on experiments related to parametric study, a mathematical model was formulated to predict the tenacity of the fiber at any spinbath composition and stretch. It was then optimized using Genetic Algorithm to get the best condition.

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CHAPTER 1

INTRODUCTION

Cellulose, found in plant walls, is the most abundant raw material on the earth. Millions of tons of this bio-renewable polymer are produced every year. Cellulose is capable of producing a number of fibrous products with excellent properties whose utility extends into numerous end uses and industries. It is an excellent source of textile fibers, for both the commodity and the high-end, fashion-oriented markets. Cellulose does not melt and does not dissolve readily in ordinarily available solvents because of the strong intermolecular bonds. To dissolve it and to utilize it for fiber making, the viscose making process was developed. In this process, cellulose is converted into sodium cellulose xanthate (through series of chemical and physical reactions using NaOH and CS_2), which is soluble in a caustic solution, making it possible to wet-spin the polymer into a fiber or film. Present thesis aims at optimizing this process to minimize the production cost. The study was conducted at Pulp and Fiber Innovation Center (PFIC), Taloja of Grasim Industries Ltd. The overall process is as follows.

1.1 Steeping:

The objective of the steeping process is to convert the cellulose to its alkoxide derivative (alkcell). In this process the pulp is added to a vigorously agitated tank of lye (17-19 %).This causes the pulp to swell up and disintegrate into small parts. However, the extent of conversion is hampered by the compact nature of the cellulose sheets, and by the accessibility of the soda into the discrete cellulose fibers. A further objective is to remove the undesirable short-chain materials present in the pulp (hemicellulose and g-cellulose), as these materials will otherwise consume CS_2 at xanthation and potentially deteriorate fiber quality. The temperature for the steeping is kept around 45 - 55 °C with the residence time of around 20 min.

1.2 Pressing:

After the pulp has been swelled and steeped in the previous step, the slurry is then pressed for the removal of excess soda. The slurry is drained into a cylindrical vessel where it is pressed hydraulically at over 120 bars of pressure. This removes all the excess soda present in the slurry to flow out of the filter plate. This lye is sent back for recirculation and can be used again after makeup.

1.3 Shredding:

Following pressing, the alkcell composition is typically 30–36 % cellulose and 13–17 % soda. Although now present as a crumb, the alkcell is relatively dense. To assist the subsequent mercerizing (pre-ageing) and regenerated cellulose fibers xanthation reactions, shredding is usually performed at this stage to open up the alkcell and facilitate the penetration of oxygen and CS_2 into the alkcell.

1.4 Mercerizing:

As received, pulp typically has a Degree of Polymerization (DP) of 750–850. For commercial viscose processes, this must be reduced to the point at which viscose dope of an acceptable viscosity will result, while maintaining the final fiber DP high enough to achieve acceptable fiber tensile properties. For regular staple production, the DP of the alkcell going to xanthation needs to be around 270–350.Typical mercerizing time ranges from 0.5–5 hours at temperatures of 40 – 60 °C.

1.5 Xanthation:

The cellulose obtained from the wood pulp is insoluble in lye. Thus, the mercerized alkcell is reacted with CS_2 vapor to produce sodium cellulose xanthate. It is this derivative which is soluble in dilute caustic soda, and when dissolved forms viscose dope. The reaction is performed under vacuum. The time for complete xanthation depends on temperature and target CS_2 level, and typically lies between 0.5 and 1.5 hours. Xanthation vessels are often jacketed to ensure constant temperature (typically 25 – 37 °C).

Sodium cellulose xanthate formation occurs as follows,

$$\text{Cellulose–O}^{-}\text{Na}^{+} + CS_2 \longrightarrow \text{Cellulose–OCS}_2^{\ -}\text{Na}^{+}$$

1.6 Dissolution:

To form the viscose solution, xanthate must be dissolved in dilute sodium hydroxide solution of the required concentration to give the final target viscose composition in terms of percentage cellulose and sodium hydroxide in viscose. Temperature plays a vital role in this process. The dissolution temperature is directly related to the kW of the dope. Lower temperatures are desirable, as the xanthate has better solubility in NaOH at lower temperatures. Forced cooling of the dissolving soda down to 0–5 °C allows better dissolving and can reduce CS_2 usage.

1.7 Viscose ageing:

Viscose dope must be aged before spinning can take place to allow for the distribution of CS_2 evenly on the cellulose chains. Even distribution is vital if stable spinning and good fiber properties are to be achieved. This ageing allows for the formation of more stable C6 after the rearrangement of C2 and C3 derivative.

1.8 Filtration:

Regardless of how well the xanthate is brought into solution, there will always be particulate material in the viscose. This type of impurity needs to be substantially removed prior to spinning to prevent blockage of the holes in the spinning jet. To filter the dope obtained after ageing, plate and frame filter press is used. The filter is operated at around 3 - 4 bar. Apart from the plate screen, filter cloth is also employed for the process. This gives the filtration range of about 10–20 μm. Care is taken to keep the filtrate cool to avoid degradation of the dope.

1.9 Deaeration:

To ensure continuity at spinning, the viscose must be deaerated to remove any dispersed air or other gases that might otherwise cause small bubbles to form as the viscose is extruded into filament form through the jet. For this process, the dope is kept in a stirring vessel while vacuum is applied across it. This increases the surface to volume ratio and helps in Deaeration:

1.10 Spinning:

The sequence of physical and chemical transformations taking place at spinning is extremely complex and still not fully characterized. The main reaction is reformation of cellulose from sodium cellulose xanthate by the action of sulphuric acid.

$$2\ \text{cellulose–O}^{-}\text{CS}_2^{+}\text{Na} + \text{H}_2\text{SO}_4 \longrightarrow 2\ \text{cellulose–OH} + \text{Na}_2\text{SO}_4 + 2\text{CS}_2$$

The spin bath also contains the sodium sulphate salt and zinc sulphate. The primary role of sodium salt is to provide a chemical potential for the water present in the cellulose to flow out. This gives fiber of better strength. The zinc on the other hand, forms a complex called zinc cellulose xanthate with the dope. This complex is responsible for the crystallinity of the fiber. The more the crystallinity, the better the tensile property. It also helps in skin effect of the fiber which is very important for the fiber to retain its dye. Immediately after the formation of this complex, the zinc forms a protective layer around the fiber which is made of compounds like ZnS and $Zn(OH)_2$. Between the formation of the zinc complex and the zinc protective layer, the acid penetrates into the fiber causing its regeneration and gives the fiber its primary structure.

1.11 Stretching

To achieve acceptable tensile properties particularly for textile end-uses, the fiber must be stretched during or very soon after extrusion. To apply the stretch, the filaments are run over godet rollers at a slower speed than the final traction units. Some stretch is also applied in the spin bath where a significant speed differential exists between extrusion velocity and take-up speed at the godet. Stretching helps in the orientation of the crystalline structure of the fiber. A better oriented fiber has higher tenacity.

CHAPTER 2

BACKGROUND

The viscose process has been around for over a 100 years. Since the time of its development, it has undergone continuous changes improving over time. The commercial viability of the process has made it an area of interest for all the major fiber and textile companies. This has led to enormous research in this field. Right from the lye concentration to the final treatment of the fiber, much work has been done to improve the process. Pertaining to the focus of this thesis, this chapter outlines some of the major works that has been done in this field.

2.1. Viscose process

Viscose process has been area of interest for lot of researchers as well as commercial industries. Since it is a widely used process for manufacturing of fiber, companies have conducted extensive research to improve and optimize it. Asahi chemicals is one such company which has extensively studied the process and filed numerous patents [1-3] on it. Details of the process have also been mentioned by Calvin et. el. [4-6]. Steeping, an important phenomenon in the process has grasped the interest of researches for long. In [8-11] the solubility of various pulp and effect of soda concentration has been described elaborately. The effect of steeping concentration on the clogging filter constant (kW) has also been studied been studied. The concept of double steeping has also been explored and several patents have been filed for the same [12]. In one of the patent, the pulp is first steeped in standard solution of alkali and then it is pressed and shredded. It is then aged and then steeped again in lower concentration alkali followed by pressing and shredded. In another patent, the pulp is steeped in normal alkali concentration. The excess lye is then drained and then additional lye of different lye is added before steeping further. In another patent, the lye is drained after steeping followed by addition of water before further steeping. All these process has had significant effect on the cost and quality of fibers. It was observed that the CS_2 consumption was decreased significantly.

Solvents other than lye has also been a subject of interest for a long time. NMMO is one of the major alternative solvent being used in the industry. In some new developments, use of urea and zinc oxide has found applications in the dissolving of cellulose.

2.2. Filter clogging constant

Filtration is an important factor in the viscose making process. If the dope is not filtered properly, the undissolved alkcell and other impurities can choke the spinneret and hinder the spinning process. The fibers obtained will also not be of supreme quality. Hence it is important to know the level of impurity in the dope. The impurity is measured in terms of filter clogging constant or kw. This filter clogging constant had been derived by Herman's and Bredee (1935) in their work on laws of filtration [13]. Application of this law resulted in the determination of impurity in the viscose dope. In [14], the method for viscose filter resistance detection has been described. The calculations are based on Hermen's and Breede filtration law. A similar method has also been patented in [15]. The time required for the dope collection vary slightly in these patents. However, [14-15] do not account for the viscosity of the dope. In [16] the viscosity of the dope has also been accounted in the form of reduced filtration value. In all the methods [14-16], the basis for the method is same. It is the method of taking the reading that varies. The equipment used for the experiment has been described in [17]. It elaborates on the design and method of the filter resistance detection equipment (kW equipment).

CHAPTER 3

MATERIAL AND METHODOLOGY

Viscose process is a very sensitive process and several authors have spent considerable time understanding and improving this process. This study aims to quantify and optimize the contribution of the spinning parameters towards fiber tenacity. The spinning process is very sensitive to the temperature. Therefore a heat exchanger was designed indigenously to control and monitor the temperature of the spinbath. The effect of the composition of the spinbath has been studied by varying each component individually and two components at a time at varying stretches. The obtained experimental data is then used to formulate a mathematical model to predict the tenacity at any given condition. This model is then optimized for maximum tenacity using Genetic Algorithm.

3.1 Standardization of equipment

The filter clogging constant (kW), which is used to determine the filterability and the level of impurity in the dope is usually calculated in a standardized big apparatus.

However that used to lead to large quantity of dope loss. Thus it was desired to standardize the small apparatus to reduce the dope loss. Several trials were conducted on big and small apparatus for the same dope.

The dope was prepared by standard viscose preparation method with standard additives as used by the Grasim industries. The pulp was acquired from cloquet. The NaOH and CS_2 used were of commercial grade.

The arrangement of the setup requires poplin cloth, filter media, perforated plate, metal gasket and a 30 µm mesh. The arrangement and the setup is shown in the fig.1. The setup comprise of a large metallic hollow cylinder capable of withstanding high pressure. The cylinders are covered with caps on both the ends. The cap on the top has the opening for air pressure and the bottom has the outlet for the filtered viscose dope to flow out. The filter assembly is fixed at the bottom of the cylinder and the dope is then filled from the top at 20 °C. The top cap is then placed and the equipment

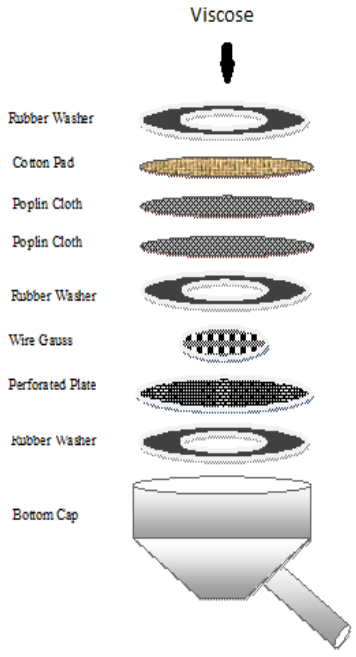


Figure 1: Kw equipment arrangement

is pressurized through air at a constant pressure of 2.1 bar. The dope is then collected in a beaker and its weight is measured at regular intervals of 10 min.

A smaller equipment of the same arrangement was fabricated. The size of the opening in the metal gasket was reduced considerably. This reduction was accounted for in the kW calculation.

The calculation for the kW:

$$\text{Kw}= \left\{\frac{T1}{W1}-\frac{T2}{W2}\right\} X2X100000X \frac{1}{(T1-T2)} \quad (1)$$

For the small equipment, the weight of the collected dope was multiplied with a correction factor. This correction factor was based on the ratio of the area of metal gasket opening.

$$\text{Correction factor}= \frac{Area\ of\ large\ equipment\ gasket}{Area\ of\ small\ equipment\ gasket} \quad (2)$$

Formula for the kW on small setup:

$$\text{Kw}= \left\{\frac{T1}{W'1}-\frac{T2}{W'2}\right\} X2X100000X \frac{1}{(T1-T2)} \quad (3)$$

3.2 Optimization of spinbath

Several parameters of spinning process contribute to the tenacity of the fiber. In this study, it has been tried to optimize the spinbath composition and the percentage stretch on the fiber. The experiments were conducted with different composition of spinbath and then the results were used to optimize the parameters using Genetic Algorithm.

3.2.1 Effect of spinbath composition

1. Design of heat exchanger:

The spinning of the dope is very sensitive to temperature. Thus it is crucial to maintain the temperature of the spinbath tray. In order to do so, a heat exchanger had to be designed as per the tray specifications to control and maintain the temperature of the spinbath.

To design this exchanger, first the heat load was calculated.

$$Q = mc_p\frac{\Delta T}{\Delta t} \quad (4)$$

$$= 1950 \text{ watt}$$

$$= UA\Delta T \quad (5)$$

$\therefore$ A= 0.1950 watt

L= 5.17 m

The length was then checked against the pressure drop to compare with the power of the pump. Pressure drop was calculated using Darcy's equation

$$\Delta P = f_d \frac{L}{D} \frac{\rho V^2}{2} \quad (6)$$

$$= 130781 \text{ Pa}$$

Then pressure drop was then checked against the pump power.

$$P_h = \frac{\dot{q}\Delta P}{3600} \quad (7)$$

2. The spinbath contains Sulphuric Acid, Zinc Sulphate and Sodium Sulphate. In order to study the effect of these three components, different baths were prepared varying each component. Also, experiments were performed varying acid-zinc and acid-salt composition. The viscose dope prepared by standard dope making procedure, was spun through these baths. The spinbath temperature was kept constant for all the trials. The temperature of wash bath was also kept constant. The stretch of the fiber was varied by changing the speed of godet and rollers. The speed of extrusion was kept constant to obtain the desired fiber denier.

3.2.2 Optimization of spinbath using Genetic Algorithm

The optimization using Genetic Algorithm was done as:

1. Formulation of mathematical model: It was assumed that an equation with 4 parameters, namely acid, zinc, salt and percentage stretch would fit into a nonlinear form and give a correlation with the tenacity. For this the equation of the form $ten=c_1 \times A + c_2 \times A^2$ was used where A=f (acid, zinc, salt, % Stretch) and c_1 and c_2 where found by minimizing the sum of squares of the error/non-linear regression.

But the variation of the tenacity from batch to batch was very high and hence wasn't generating accurate results. It was evident from the experimental data that the same condition was not yielding same tenacity for different batches. In order to eliminate this problem, the base condition was taken as zero in every batch and the other data points were normalized with respect to the base condition for that particular batch. In this way the normalized data points were obtained for all the batches and those were used to model the system.

However, the difference between the base cases of the batches from which the data has been taken is very large. This has given rise to an error of the range of ±0.18.

2. Optimization using Genetic Algorithm: After the modelling of the system, it was desired to determine the condition which maximizes the tenacity. Hence a code was written in Matlab with tenacity as a function of the 4 parameters (acid concentration, zinc concentration, salt concentration, % stretch). GA solver was used specifying the bounds of the parameters. All the rest option were kept at default.

Genetic algorithm, however, only minimizes any given function. In order to maximize the function f(x), a new function F(x) was defined such that $F(x) = \frac{1}{(1+f(x))}$ and then F(x) was minimized.

CHAPTER 4

RESULTS AND DISCUSSION

The experimental setup of the kW equipment has been dealt in detail in the previous chapter. The standardization helped in reducing the amount of dope being wasted during the experiment. The process and parameters details of the spinning experiment has also been explained elaborately in the last chapter. The observations and the results of these experiments are of utmost importance to this study. These results and observations have been discussed in details in the following chapter.

4.1 kW standardization:

The kW standardization involved a number of trials of the same dope on big equipment as well as the small equipment. The readings for both the equipment were compared and the correction factor was fine tuned to standardize the equipment.

It was observed that

1. The viscose dope flow rate per unit area per unit time was decreasing faster in the small equipment than the big equipment. This can be seen in fig. 2 (a). The small equipment flow rate is flattening out faster than the big kW. The flowrate has been higher since the beginning of the experiment. At time 5 min, the flow curve of the big equipment has a flowrate of 2 whereas that of the small one has 0.7. The difference between the flowrate curve increases as the time proceeds and at 40 min, big setup kW has a flowrate reading at 6.5 and small setup has it at 3.1. The small kW flowrate is increasing almost linearly while that of big setup is concave to the origin.

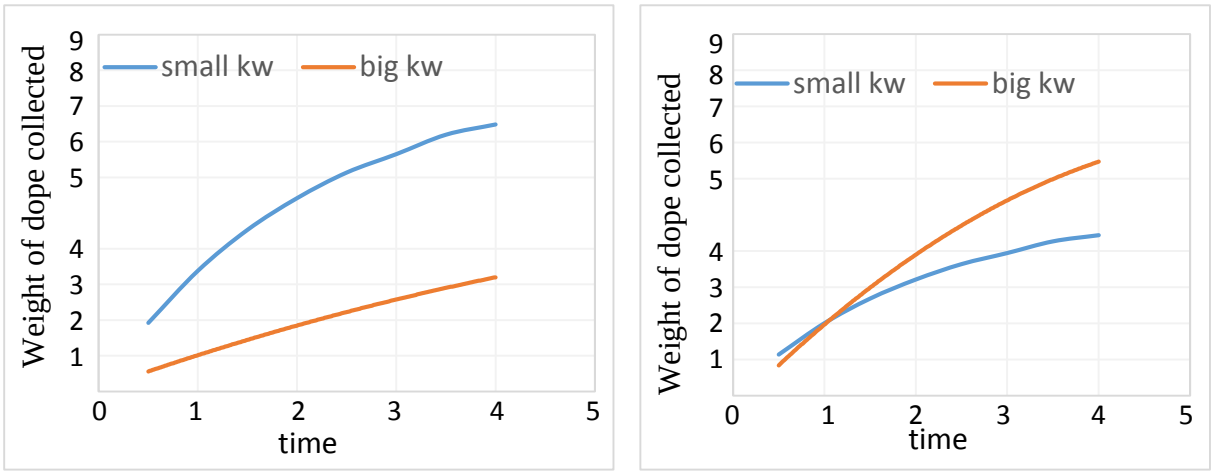


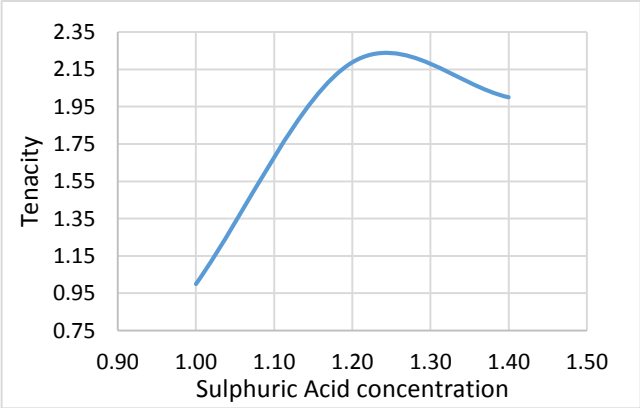
Figure 2: plot of weight of dope collected vs. time when (a) kW was matched in both the equipment (b) flowrate was matched in both the equipment

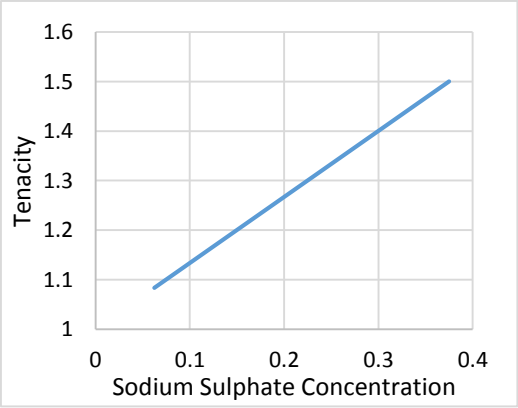
2. Also, when the correction factor was so adjusted to match the kW values of the two equipment, the flow rate curve of the two equipment were not matching as observed in fig. 2 (a) the curves had a significant difference between them.
3. When the correction factor was so adjusted as to overlap the two flow rate curves as much as possible (fig. 2 (b)) the kW reading obtained from small equipment had a large variation compared to the large one. We can see in the figure, the small kW curve starts above the big kw curve at 5 min, they intersect each other at 10 min and then the gradually, the distance between the two curves widens as the small kw starts slopping downward.

4.2 Optimization of spinbath

4.2.1 Effect of spinbath composition

Different components of the spinbath contributed differently to the tenacity of the fiber and showed different trends for the same. At a constant stretch, when the acid content was varied, it was observed that the tenacity increased with increase in the acid. When the acid content in the spinbath was increased, tenacity was observed to have increased at the same stretch. We can see in Figure 3(a), as the acid is increased from 1 to 1.23, the tenacity increases from 0.96 to 2.20. But after acid concentration of 1.23 till1.40, the tenacity decreases to 1.96. On the other hand, salt had an almost linear effect on the tenacity of the fiber (Figure 3(b)). The tenacity of the fiber increases from 1.11 to 1.5 as we increase the salt from 0.06 to point 0.37. Effect of change of zinc concentration in the spinbath can be seen in Figure 3(c). The tenacity increases with the increasing zinc, peaks and then decreases. As we zinc concentration increases from 1 to 5, the tenacity also increases from 0.35 to





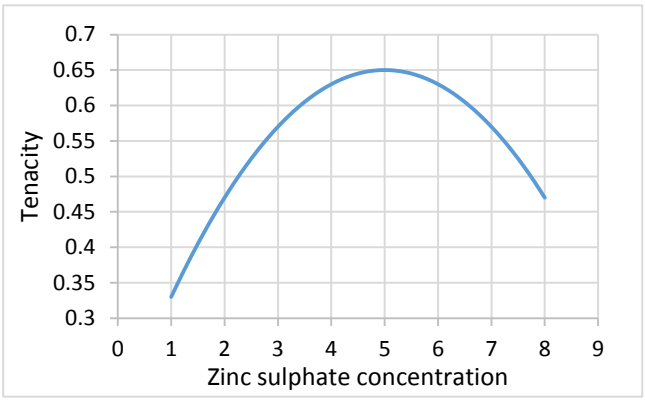


Figure 3 : Tenacity vs spinbath components at constant stretch (a) tenacity vs acid concentration (b) tenacity vs salt composition (c) tenacity vs zinc composition

0.65. It attains the maximum value and then starts decreasing thereafter. It comes down to 0.46 at a zinc concentration of 8.

The increase in stretch in all the compositions had more or less the similar trend. The tenacity increases with increase in stretch and then decreased after a peak. When we look at the fig. 4(a) for varying acid, we notice that for base case, the tenacity is increasing from 1.5 to 5 as the stretch is increasing from 0 to 3. However, after that the tenacity becomes almost constant from 2 to 2.2 stretch. After that the fiber breaks. In the case of 1.16 acid, the tenacity increases from 1.1 to 5.1 as the stretch increases from 1.4 to 2. It reaches the peak at 2 and then starts declining as the stretch is increased from 2 to 3. The fiber then breaks at 3. For 1.3 acid case, the tenacity increases from 0.4 to 4 as the stretch is increased from 0.4 to 2.5. The tenacity peaks at 4 and then starts declining as the stretch is increased to 3 after which the fiber breaks. For the case of zinc sulphate, (fig. 4(b)), if we look at the base case with zinc-1, we observe that the tenacity increases quite steadily from 3to 4.9 as the stretch is increased from 0.3 to 1. It then flattens at a bit and increases from 4.9 to 6.4 as the stretch is increased from 1 to 2.5. For the case where zinc concentration is 2.5, the tenacity increases from 2.7 to 5.1 steadily as the stretch is increased from 0.4 to 2. It then peaks at 5.5 at a stretch of 2.2 and then starts decreasing. It comes down to 4.2 from 5.5 as the stretch is increased from 2.2 to 3. The fiber then breaks. When Zinc concentration is 4, the tenacity increased almost linearly from 1.1 to 3.1 as the stretch is increased from 0.4 to 2. It then reaches a peak and then starts decreasing from 3.1 to 2.1 as the stretch increases from 2 to 2.45. The fiber then breaks. In case of salt with base case condition (salt-1) in fig. 4(c), the tenacity does not seem to be affected much by the stretch. It is almost constant at 0.9 as the stretch is increased from 1.8 to 5. It then increases slightly to 1.2 as the stretch is increased to 6. Increase in salt has yielded better tenacity. If we look at the case with salt concentration 1.6, we see considerable increase in the tenacity at

1.8 stretch when compared to the base case. It has a tenacity of 2 which increases to 3.4 and reaches the peak as the stretch is increased from 1.8 to 5. It then starts decreasing from peak to 2.9 at stretch

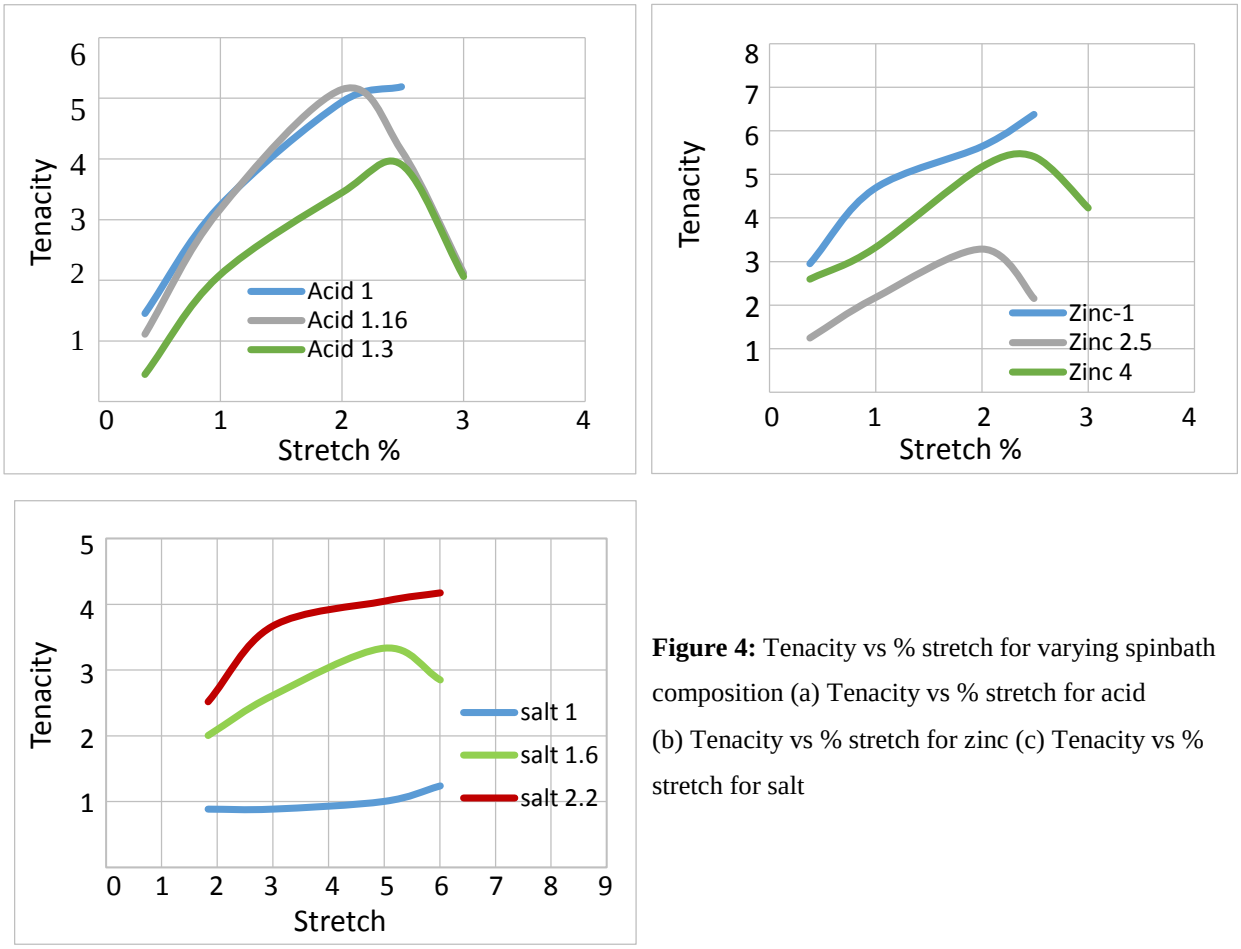


Figure 4: Tenacity vs % stretch for varying spinbath composition (a) Tenacity vs % stretch for acid (b) Tenacity vs % stretch for zinc (c) Tenacity vs % stretch for salt

6 and then breaks. In the curve depicting highest salt concentration of 2.2 it can be observed that the tenacity increases from 2.5 to 3.7 as the stretch is increased from 1.8 to 3. The tenacity thereafter is increasing at very slow rate. It increases to 4.1 as the stretch is increased to 6 after which the fiber breaks.

In the experiments where 2 parameters where varied together, interesting observations were made. When acid and zinc was varied together, it was observed that the tenacity decreased with increasing acid and zinc content (Figure 5 (a)). For the base condition of acid-1 zinc-1, it was observed that the tenacity increased steadily from 2 to 5 as the stretch increased from 0.4 to 2. It reaches the peak and then starts decreasing as the stretch is further increased. The highest stretch achieved was 2.5 at which the tenacity was 4.5. For the case where composition is acid-1.16 zinc-2, the tenacity was 2 at the stretch of 0.4. It increased to 3 as the stretch was increased to 0.8 after which the tenacity

was almost constant till stretch 1.8 after which it started decreasing rapidly. It reduced to 2.2 at a stretch of 2.5. For the highest case composition of acid-1.3 zinc-3, not high tenacity was observed. At the base stretch of 0.5, a tenacity of only 1.1 was observed. It increased steadily to 2.7 as the stretch was increased to 2. After which it peaked at 2.8 for a stretch of 2.2. The tenacity started to fall after that and reached 2.2 at a stretch of 3. The fiber broke after that.

The effect of acid-salt was quite opposite to that observed in acid-zinc combination. Increase in the amount of acid-salt in the spinbath resulted in increase in tenacity (Figure 5(b)). If we look at the base composition of acid-1 salt-1, the tenacity increased from 1.5 to 3 as the stretch increased from 0.4 to 1. After that the increase in the tenacity was not very high. It reached 3.5 at a stretch of 2 before breaking. For the composition acid-1.16 salt-1.3, tenacity of 2 was observed at a stretch of 0.4. It increased steadily to 6.7 as the stretch was increased to 2. The fiber reached peak tenacity there and then started to fall.

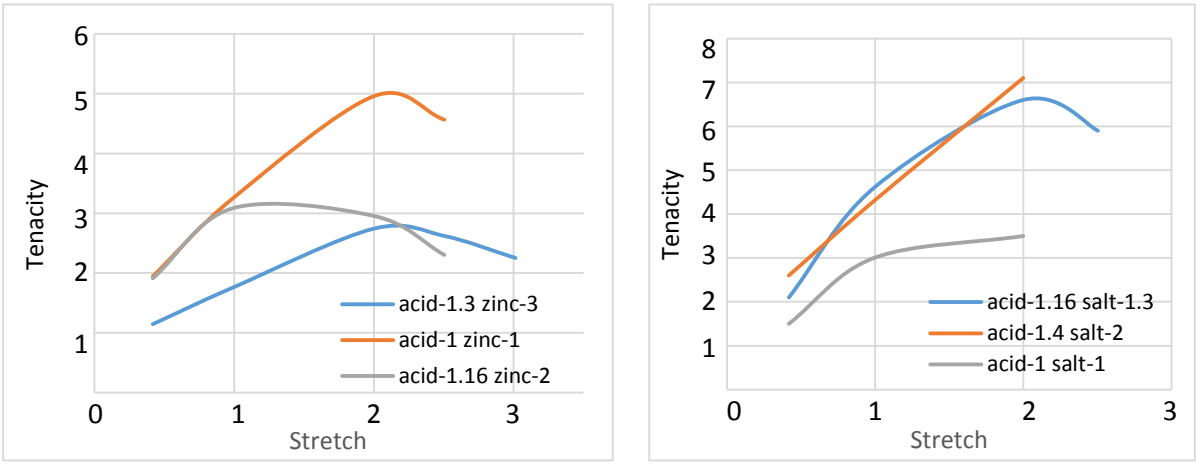


Figure 5: Tenacity vs. stretch plot with varying 2 components at a time (a) Tenacity vs. stretch with varying acid-zinc (b) Tenacity vs. Stretch with varying acid-salt

4.2.2 Optimization of Spinbath using Genetic Algorithm

The mathematical model was prepared with as many as 36 data points in order to increase the goodness of fit. But since the base case for all the data points were different, and the difference between the base cases were very high, there exists an error of ±0.18 in the results. However, it can be confidently said that the model shows very accurate directional trends. The mathematical model formulated gives the tenacity at any given condition with acid, zinc, salt and % stretch as the parameters.

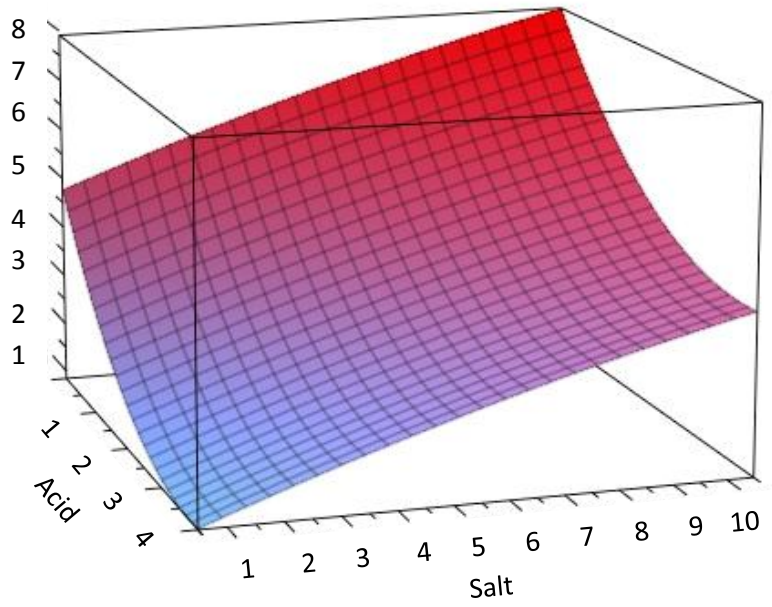


Figure 6 : acid-salt-tenacity plot

The mathematical model was plotted in 3 dimensions with 2 independent variables at a time. Varying acid and salt was plotted for fixed base concentration of zinc and stretch percentage (Figure 6). It was observed that at lower salt, as the acid is increased, the tenacity decreases. At acid 1 and salt 1, the tenacity is close to 4.6. But as the acid is increased to 4, the tenacity decreases to almost zero. Similar trend is observed when salt is increased. At salt 10, when the acid is 1, the tenacity is 8. But as the acid is increased to 3, the tenacity decreases constantly. It then stays almost steady as the acid is further increased to 4.

In the plot varying zinc and acid vs tenacity, where salt and stretch percentage is kept constant, it was observed that the tenacity increased, reached a peak and decreased with increasing zinc at a constant acid. At acid 4, the tenacity increased from 1.6 to 2.5 as the zinc increased from 0 to 2.2. The tenacity then decreased to almost 0 as the zinc is increased to 4. As the acid is decreased, the same trend is observed. At acid close to 0, the tenacity is 5.1 as zinc reaches 0. The tenacity then increases to 6 at zinc 2. It then decreased to 4 at zinc 4.

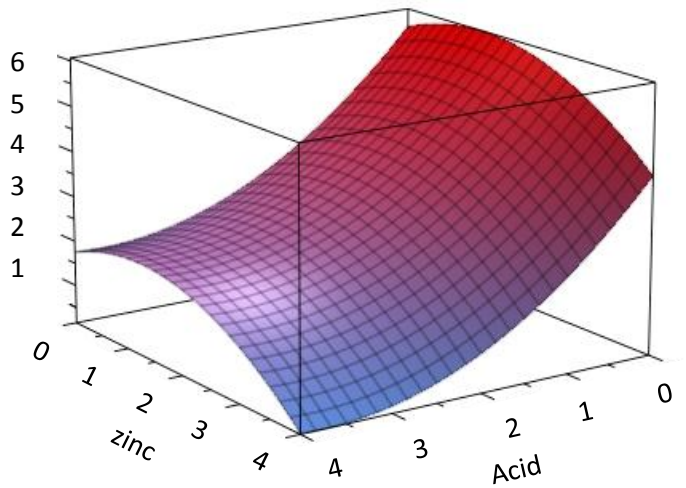


Figure 7 : zinc-acid-tenacity plot

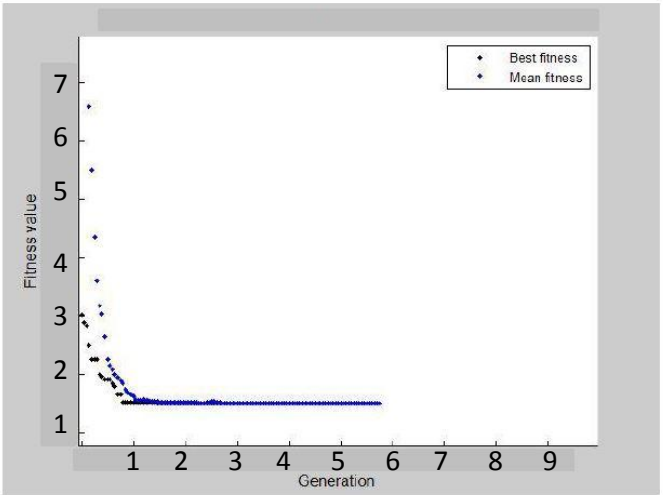


Figure 8: fitness value vs generation plot

The optimization of the model using genetic algorithm gave the condition of maximum tenacity at 33 % lower acid, 10% higher zinc, 30 % higher salt and 47% higher stretch than the base case for the optimum value. It also predicted the maximum tenacity value achievable which was between 20-30% higher than the base condition. As it can be seen from Figure 8, the fitness value starts near 7 at the generation close to zero. It then drops to 1.5 at the 1st generation and becomes constant. The algorithm runs till almost 6th generation and then terminates as the tolerance is reached. The system was not very stiff and converged smoothly to the best value.

CHAPTER 5

CONCLUSION

1. The aim of the thesis was to improve the tenacity of the fiber by changing the process parameters and process conditions. The focus of this objective was towards the spinning side. But the dope preparation was also an essential part of the process. To get maximum quantity of dope without compromising the quality, it was important to standardize a small kW equipment. To do so, a small setup equipment was fabricated and rigorous trials were conducted on the standard as well as small setup kW equipment. The readings of the small setup equipment were multiplied by a correction factor and then the kW was calculated. This correction factor accounted for the change in the area of the opening of the equipment through which the filtered dope is obtained.

However it was observed that the flow rate profile of the two equipment were not matching despite the use of correction factor. The small equipment flow rate curve was flattening out faster than the big equipment. Also, when the kW of the two equipment were matched by adjusting the correction factor, the flow rates curves were not touching/overlapping each other.

Further study could be conducted to determine the cause of the difference in the flow rate. The reason for the difference in the flow rate profiles is also not very clear and could be an interesting area to explore further.

2. The composition of spinbath was optimized by conducting experiments and optimizing the results using Genetic Algorithm. But the viscose process is highly sensitive to the spinbath temperature. Hence it was desired to conduct the experiments at a controlled temperature. A heat exchanger was designed, therefore, for the spinning tray to regulate the temperature. The design included calculating the heat load and the length of the exchanger for the given pipe diameter, and finally the pressure drop for the same to check against the motor requirement. Then several trials were conducted varying the spinbath composition. These included varying one component at a time and two components at a time. This gave sufficient data points to formulate a mathematical model by regression to predict the tenacity by defining the composition of the spinbath and the % stretch. Further, the model was then optimized using genetic algorithm to maximize the tenacity and obtain the best condition for that.

The model was prepared using the data generated by varying the spinbath composition in some range. The accuracy of the model beyond that range is not very conclusive and can be a subject of further study.

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