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OPTIMAZATION AND RECOVERY OF CAUSTIC SODA FROM THE MERCERIZATION PROCESS: ALMEDA TEXTILE FACTORY

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ADDIS ABABA, ETHIOPIA

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Optimization and Recovery of Caustic Soda from the Mercerization Process: Almeda Textile Factory

A thesis in partial fulfillment of the requirements for the degree of Master of Science in chemical engineering under the stream of process engineering

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DECLARATION

I, the under signed, this thesis or any part of it has not been submitted elsewhere for the award of any degree or diploma. I have dually acknowledged and refereed all materials used in this work.

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ABSTRACT

An experiment was conducted to evaluate the operational parameters of an evaporation unit which concentrate dilute caustic soda from the mercerizing operation wastewater unit of a textile mill. The concentration of caustic soda for the mercerizing operation was optimized before recovery and optimum concentration of 20-23% was attained. In this process, the concentration of caustic soda in the wastewater was raised to the optimized range 20-23 w/w % by evaporating the water from the dilute solution collected from the mercerizing effluents which contain 5 w/w% caustic soda. The process unit used for this purpose consists of an evaporator, separator, steam generator, condenser, feed vessel, storage tank and vacuum pump. Parameters including the amount of mass condensate, the amount of mass concentrated, temperatures, evaporator pressure, heating steam supply pressure and concentration change of caustic soda solution were measured. From these, the rate of evaporation, and concentrating capacity of the evaporation were determined. From the experiments, it was found that the rate of evaporation ranged from 2.24 kg/hr to 6.41 kg/hr with an average of 5.71 kg/hr.

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Symbols and Indices

Symbols:

Q	energy (kJ)
m	mass(kg)
$\dot{m}$	Mass flow rate (kg/s)
P	Capacity / kg heating steam
p	Pressure(bar)
t	Time (s)
f	Flow rate (L/hr)
ρ	Density (kg/m^3)
T	Temperature (°C / K)
E	Energy (kJ)
h	Specific enthalpy (kJ)
P_3	heating steam supply pressure
F_1	feed flow rate
P_2	Evaporation pressure (bar)

Indices:

CRS	Caustic recovery system
Tot	Total
Evap	Evaporation
Loss	Loss
Conc	Concentrate
Cond	Condensate
Heat.	Heating steam
Evapor	Evaporator
HSC	Heating steam condensate
Cool	Cooling water

1. Introduction

One of the basic necessities for human beings is Clothing. This has ultimately led to the development of textile industries that produce fibers and process them to produce cloth to meet the demands of the increasing population and the living standards of modern civilization. In Ethiopia the history of modern textile industry goes back to the late 1930's with the establishment of the first textile factory, Dire Dawa Textile. Joint venture investments with Italian, Japanese, Indian and British companies were instrumental in the early development of the textile industry(Hassen, 2008). The development of the modern textile sub-sector has made historical contribution to satisfying domestic needs of fabrics, generating employment opportunities and promoting national economic development in addition to establish the basic foundation for manufacturing industry in Ethiopia. At the same time the textile sub-sectors' product export has earned large sum of foreign currency for the country(Siyum & Kindeya, 2016).

In general, the textile processing starts with yarn formation followed by weaving and finally end with dyeing and finishing stages. Dyeing and finishing processes, termed as "wet processing" improve the quality of the fabric in terms of appearance, durability and luster. However, these processes are responsible for making the textile industry a highly water and chemical intensive industry. A typical textile facility uses 200-400 liters of water to produce 1 kg of fabric(Khanam, 2011). Consequently, large quantities of wastewaters are also generated. The chemical composition of textile effluents is changing rapidly as a result of shifting consumer preferences. Most significant is the current popularity of cotton fabrics and bright colors leading to the greater use of reactive dyes respectively. The effluent is alkaline and the usage of caustic soda in the treatment of the fabrics in a mercerizing process is one of the main contributors for this.

Mercerization is one of the major finishing processes of cotton textile production where caustic soda (NaOH) is the main chemical used. During this process, cotton yarn or fabric (mainly woven fabric, but also knitted fabric) is treated in a solution of concentrated caustic soda, generally 200-300 g NaOH/l or 170-350 g NaOH/kg of textile substrate, for about 40 - 50 seconds (Gemci, 2010). This process is generally applied to increase dye affinity, luster and strength of fiber. Wherever it is applied, mercerization process is followed by an intensive hot water rinsing or washing process to remove the excess caustic. As a result, a

highly alkaline and relatively hot wastewater which includes various other organic and inorganic impurities is generated.

The mercerizing process generates problems of wastewater with high alkaline content that leads to high amount of caustic soda losses for textile industry. The mill incurs cost arising from loss of valuable caustic soda that is not reused and also from having to neutralize this alkali with acid during waste treatment in order to meet the standards set by the government for disposal of wastewater . However, there are methods by which this caustic soda can be recovered. One such method is by evaporation of water from the dilute wastewater to concentrate it so that it can be reused again in the mercerizing process(Sharma & Sharma, 2015).

Evaporation is an accepted way of recovering caustic soda from wastewaters and has been applied in many textiles since 1900(Rughurman, 2011). Generally multiple effect evaporation processes are used. Their performance has been satisfactory in concentrating dilute caustic solution to concentrated ones. However, this is an energy intensive process. Almost all industries use steam as the heating medium to drive away water from the dilute solution. The Ethiopian government is now giving due emphasis to the textile factory. There are different governmental and private textile factories operating in the country. Almeda textile factory is one of the private limited companies that produce yarns and fabrics and it was established in 1996.

1.1 Problem statement

A textile industry plays an important role in supporting and sustaining human life around the globe. Consumptions of the products of the industry are central themes of the survival and development of Mankind. Hence, textile industries are known as evergreen industries as they will last ever since there is a need for clothing. Moreover, the textile industry can play an important role in a nation's economic development, especially in least developed countries, like Ethiopia. Experience of many countries, particularly of the Europe and Asian continent, revealed that textile and garment industries have made a considerable contribution as the base for industrial development and source of employment creation, income generation and export earnings. Both in domestic and international aspects, Ethiopia have fine development opportunity and huge potential in developing a textile industry. Available within Ethiopia are all essential ingredients for a competitive textile industry: raw materials, low wages and low energy costs. This gives the country a comparative advantage over other countries and regions. The Ethiopian Government is actively promoting the further modernization of the textile sector with the aim of attracting foreign investors that can penetrate the global market.

Ethiopian and foreign investors are recently moving aggressively in the direction of higher value addition through investing in the balancing, modernization, establishing and up-gradation of the textile manufacturing industry by acquiring new technologies and knowhow. Textile industries use substantial quantities of caustic (sodium hydroxide) to clean and prepare fabrics for dyeing. But caustic is expensive and its disposal may create a hazardous waste treatment problem. Other textile factories like European and Asian use a caustic recovery plant to overcome the waste and cost problems. But in Ethiopia (Almeda textile factory) they didn't use this very interesting technology recovery plant for caustic soda from mercerization process. Therefore, this paper is aimed to fill the gap in Ethiopia in acquiring the technology (caustic soda recovery system) for a healthier environment and to remain competitive with other foreign industries and to become benefitairy from the sector.

1.2 Objectives

1.2.1 General objective

The objective of this research is to evaluate caustic recovery from mercerizing process by evaporation with the ultimate goal of producing a caustic stream at reusable concentrations

1.2.2 Specific objectives:

1. To characterize the mercerizing effluent (caustic soda containing waste)
2. To optimize the concentration of caustic soda for mercerization
3. To evaluate the optimum performance of evaporator in caustic soda recovery in terms of rate of evaporation, and concentrating capacity

1.3 Significance of the study

Caustic Recovery Plant (CRP) concentrates weak caustic into strong caustic by passing steam through heat exchangers to evaporate excess water from the solution. At the end of the process, the concentrated caustic is recovered, while vapor from the last stage is condensed with cooling water, where it absorbs the waste heat from the vapor. This results in hot water generation as by-product, which can be used in other units. The vapor condensate is slightly alkaline soft water without any hardness. It has a temperature of approximately 90 °C(Körting , 2010). It can be used for washing, e.g. in the mercerizing or bleaching machine, or in other pre-treatment.

The CRP requires heating steam and cooling water. Almost the same amount of steam which is used for the recovery of the mercerizing lye can be saved in the hot water generation. This hot water generation is a by-product in which the cooling water is heated up to 60 °C to 85 °C. The CRP is very energy efficient, especially when hot water generation is integrated in the central hot water system. There is no direct contact between the heating steam and the lye; therefore the heating steam condensate can be reused as boiler feed water without additional treatment(Korting,2010).

Integration of the CRPs will prove to be highly effective and cost efficient for the factory with the following measure expected benefits:

- Recovery of caustic soda, which can be further reused
- Reduced chemical consumption for neutralization
- Generation of hot water as a by-product(from the waste energy)
- Minimization of alkaline wastewater from mercerizing machines
- Protection of the environment (environmental protection: fewer chemicals for Neutralization will be needed).

Generally this plant will have a great advantage in both to the factories and to the nation (Ethiopia):

- Economically competitive income from this sector (textile industry)
- Remains competitive in the world market
- Will have proudly high quality products
- Job employment of youths

2. Literature review

2.1 Textile Industry

The modern Textile manufacturing begins with the production or harvesting of raw fiber. Fiber used in textiles can be harvested from natural materials (*e.g.* wool, cotton) and manufactured from regenerative cellulose materials (*e.g.* rayon, acetate), or it can be completely synthetic (*e.g.* polyester, nylon). After the raw natural or manufactured fibers are shipped from the farm or the chemical plant, the natural or synthetic fibers will be transformed into yarn. This yarn is transformed into fabric or similar products which undergo further processing, such as dyeing and finishing to be converted into marketable textile products. Generally they pass through four main stages of processing(Wadje, 2009):

- Yarn production
- Fabric production
- Wet process
- Garment Manufacturing

2.2. Yarn formation

Yarn fabrication is the progression, which converts raw fiber into yarn or thread. The fibers are organized and then drawn out and warped to form the yarn, which is then wound onto a pin or cone. The yarn fabrication is completely dry, although some yarns maybe colored and finished as a final customer product.

The majority textile fibers are processed by spinning processes. On the other hand, processes leading to spinning are depended on the nature of fiber. Before yarn formation, fibers go through the processes of opening, blending, carding, combing and drafting(Wadje, 2009).

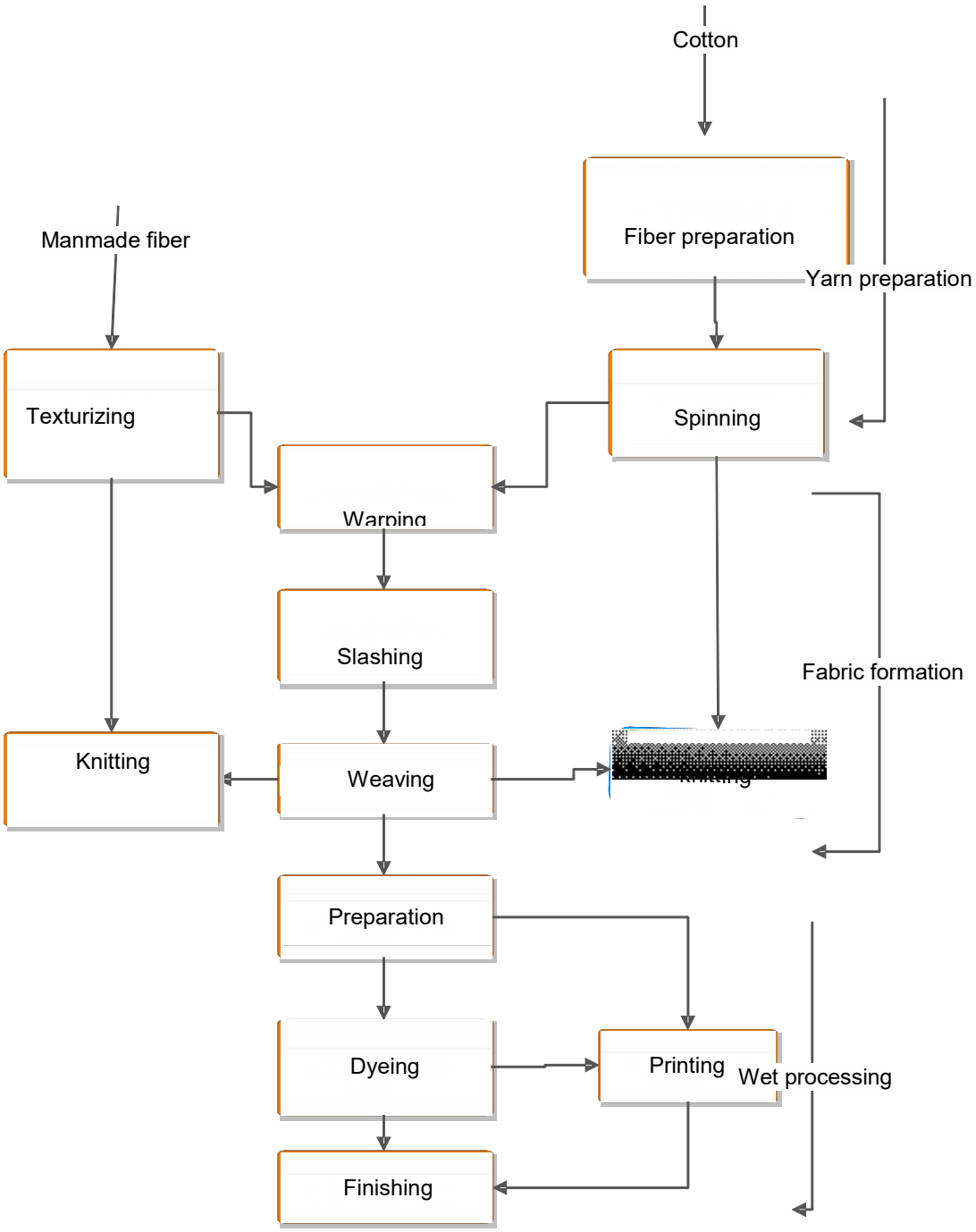


Figure 2-1 textile industry processing flow diagram

2.3. Fabric formation

Weaving and knitting are the two most important processes of fabric manufacturing. By weaving, the yarn is assembled together in a loom and a woven fabric is obtained. By knitting, process yarn is knotted together with a succession of needles (Cherif, 2016)

2.4. Wet processing

Wet processing improves the fabric in terms of appearance, durability and serviceability. Wet processes generally include four steps: fabric preparation, dyeing, printing and finishing(Cherif, 2016).

Figure 5.4 shows the steps.

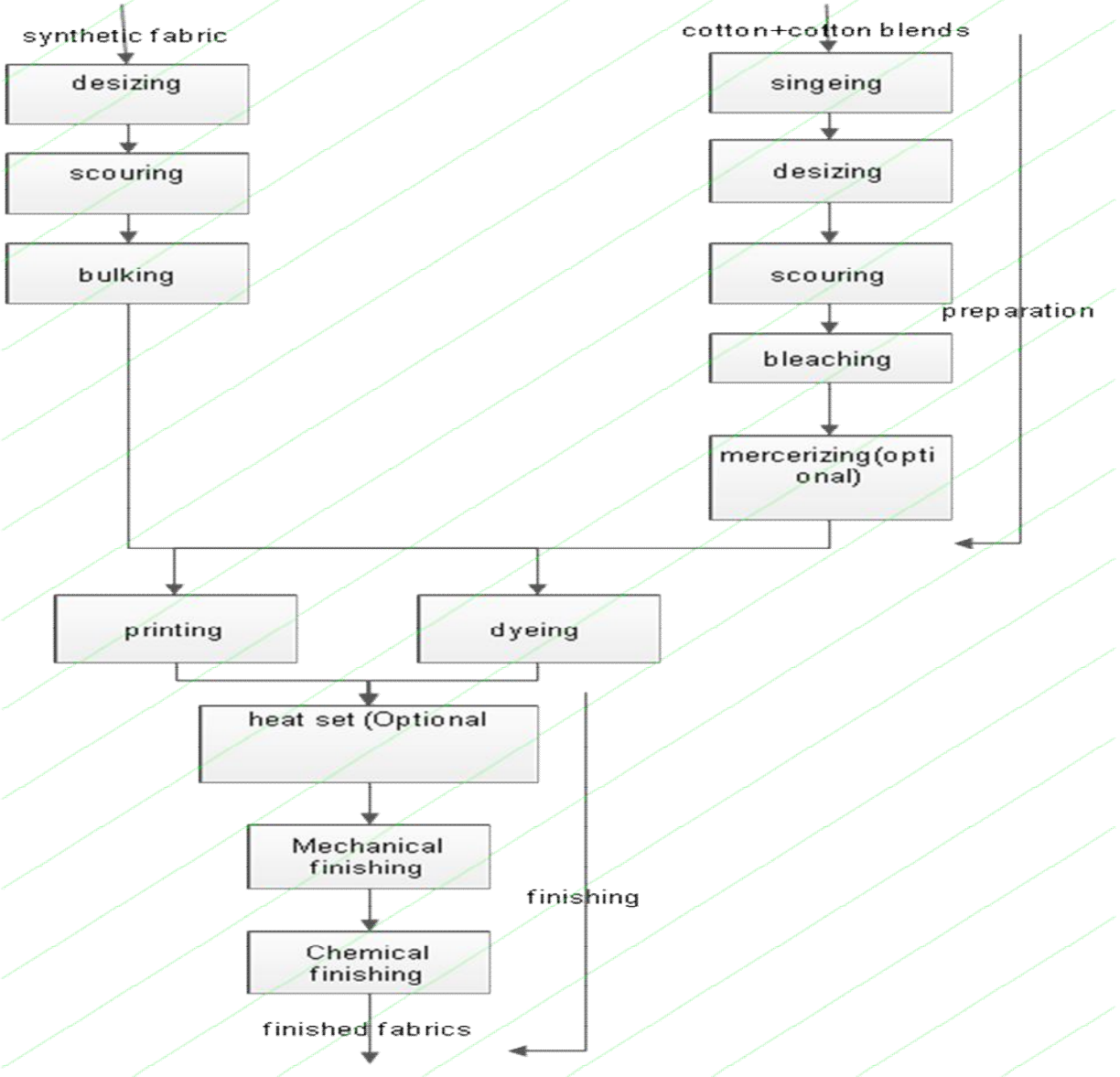


Figure 2-2 wet processing

2.4.1 Fabric preparation

This is carried out to advance the wetting capacity and adsorption, dye take-up capacity, purity of the fabric, to lighten and for improved material development. Cotton preparation follows five steps: singeing, desizing, scouring, bleaching and mercerizing (Menezes & Choudhari, 2012).

Singeing is carried out to eliminate the fiber ends protruding from the yarns or fabrics by passing them over flame or heated copper plates. Desizing is carried out to eliminate sizing materials which had been used before weaving and knitting operations. These would otherwise unfavorably react with chemicals applied at some stage in further processing. Scouring is carried out to make dirt free fabrics and yarns. Bleaching is carried out to eliminate matters that would adversely have an effect on the whiteness of the fabrics (Cherif, 2016; Menezes & Choudhari, 2012). Mercerization is the treatment of cotton with a strong caustic alkaline solution in order to get better of the luster, hand and other properties was named following its discoverer, John Mercer, and it has been in use for some time. It has been seeing raise in application recently.

The methods and effects concerned in the processing of cotton and polyester are unlike, but, both involves a treatment with strong alkaline solution before dying to improve the properties of the fibers, and so two of them can be considered together to be alkaline treatments. In recent times, there has been large use of so-called soda reduction processing, which treats polyester with a strong caustic soda solution to dissolve and remove the surface film in order to improved the hand(Khalifa, 2017).

2.5 Mercerization process

If cotton fabric is treated in a strong alkaline solution the fibers swells and shrinks. If the fiber is placed under tension while in this enlarged state and then rinsed with water, the alkali will be detached and lasting silk-like luster will result. On the other hand, after swelling, if the alkali is washed off when the fibers are in its shrunk state, to enhance in luster may not be desirable, but the fibers will secure in that shrunk state, as a result giving better elasticity to external tension. The former is known as tension mercerization and is often simply called mercerization, while latter is referred to as slack mercerization. By the reason of considerations of cost and efficiency of alkalis, only caustic soda is used as the alkali in industry.

2.5.1 The effects of mercerization

The effects of mercerization process are improves luster, increases ability to absorb dye, improves reactions with a variety of chemicals, improves stability of form improves strength/elongation, improves smoothness and improves hand. The appearance of the fabrics are improved through amplified luster, a deepening of the color and the production of a transparent look, the sense of the fabric is improved through a resulting flexible soft hand and improved smoothness, strength and elongation are as well improved, along with the addition of better stretching ability. The treatment and handling can be altered to fit different requirements, thus allowing for the most excellent appliance of the results of different processing(D Lee,2007,Khalifa, 2017) In the mercerization process, the fabric is treated with caustic soda (sodium hydroxide) solution. Mercerization can be two types, with tension and without tension. The most familiar method involves application of 200-300 g/l caustic soda solution to the cotton yarn/fabric under tension for less than 1 minute. The temperature is kept in range of 5-18 ºC since the process of caustic application itself is exothermic. The alternative method, where out tension is applied, involves application of concentration 145-190 g/l caustic soda solution at 20-30 ºC. This latter process is also known as a slack mercerizing, or caustification. Another rare common method is ammonia mercerization where cotton yarn or fabrics are treated with anhydrous liquid of ammonia(Menezes & Choudhari, 2011).

The most important parameters of mercerization process are the concentration of caustic (other parameters include time of application, tension applied, machinery and ambient temperature). The accurate amount of caustic must be applied to increase the effectiveness of the dyeing process. Nevertheless, excessively use of the caustic can lead to difficulties of washing it out in the next stages. So maintaining the appropriate concentration is essential.

2.6. Dyeing

This involves coloring of the fabric in the textile by either a batch process or a continuous process. In both processes, dye is applied to the textile and depending on the type of fiber and affinity of the dye type for the fiber; the dye molecules come into the fiber over a period of time. Supporting chemicals and operating conditions speed up the process. Sometimes, by application of heat, dye fixation is enhanced. Finally washing will be carried out to remove the unfixed dye and chemicals from the textile(Menezes and Choudhari, 2011; Wadje, 2009).

2.7. Printing

Printing is like dyeing, but differs with its restricted coloring application. Four basic steps to textile printing are dye paste preparation, dispersion of color or pigment in a printing paste (printing), application of dye or pigment to the textile, drying of fiber with a steam or hot air followed by washing (Sharma and Sharma, 2015).

2.8 Finishing

This is the final stage in the textile production and mechanical or chemical treatments are carried out depending on the end-use purpose of the textile factory. Physical treatments are brushing and ironing which helps to increase the luster and feel of textile is called mechanical finishing. Chemical treatment supports in a variety of properties such as easy care, water-repellent, to softening and flame retardant(Cherif, 2016).

2.9 Evaporation

Evaporation is the removal of solvent (usually water) as vapor from a solution or slurry. For the overpowering majority of the evaporation systems the solvent is water. The objectives are usually to concentrate a solution; therefore, the vapor is not the desired product and may or may not be recovered depending on its worth. Therefore, evaporation usually is achieved by vaporizing the portion of the solvent producing a concentrated solution, thick liquor, or slurry.

Evaporation often encroaches upon the operations such as distillation, drying, and crystallization. In evaporation, no attempts are made to separate components of the vapor. This differentiates evaporation from distillation. Evaporation is distinguished from drying in that the residues are always liquids. The desired products may be solids, but the heat must be transferred in the evaporator to the solution or to the suspension of the solid in a liquid. The liquid is usually highly viscous or a slurry. Evaporations are differ from crystallization in that evaporations are concerned with concentrating the solutions rather than producing and building crystals(Shah and Bhagchandani, 2012).

2.9.1 Types and principles of evaporation

In the field of thermal separation (concentration technology), evaporation plants are widely used for concentration of liquid in the form of solution, suspension, and emulsion. The major requirement in the field of evaporation technology is to maintain the quality of the liquids during evaporation and to keep away from damage to the product. This may require the liquid to be uncovered to the lowest possible boiling temperature for the shortest period of

time(B.Glover, 2004). These and numerous other requirements and limitations have resulted in a wide variation of designs are available today. In almost in all evaporators the heating medium is steam, that heats a product on the other side of a heat transfer surface. The following lists contains the descriptions of the most common types of evaporators(Getzville, 2008.).

1. Falling Film Evaporators
2. Rising Film Evaporators
3. Forced Circulation Evaporators
4. Plate Evaporators

2.9.2 Falling film evaporators

In falling film evaporators, liquid and vapors are flow downwards in parallel flow. The liquid to be concentrated is preheated to boiling temperature. An even thin film enters the heating tubes via a distribution device in the head of the evaporator, flows downward at its boiling temperature, and is partially evaporated. This gravity-induced downward movement is increasingly augmented by the co-current vapor flow. Falling film evaporators can operate with very low temperature differences between the heating media and the boiling point of the liquid, and they also have too short product contact times, typically just a few seconds per pass. These characteristics make the falling film evaporator principally suitable for heat-sensitive products, and it is today the most frequently used type of evaporator because of its application(B.Glover, 2004).

2.9.3 Rising film evaporators

A rising film evaporator is used for evaporating or concentrating solutions. The unit consists of the actual rising film evaporator for evaporation of the feed, a cyclone separator for separating the vapour phase from the liquid phase. This type of evaporator is often used with product recirculation, where some of the formed concentrate is recycled back to the feed inlet in order to produce sufficient liquid loading inside the boiling tubes(Salakki et al, 2014).

The rising film evaporator is a stainless steel double pipe heat exchanger heated by steam. A temperature sensor at the head of the evaporator is used to measure the temperature of the vapour of the feed. A cyclone separator allows the separation of the liquid and vapour phases to be observed. Separation occurs because of the centrifugal forces resulting from the incoming steam in the cyclone. The vapour phase emerges at the head of the cyclone and is fed into the condenser. The concentrated liquid accumulates in the bottom section of the cyclone and flows into the concentrating tank.

The concentrate can be continuously drawn off using the concentrate valve or fed back into the rising film evaporator by the return valve. The temperature sensor records the return temperature during the operation. The return pipes are made of non-corrosive stainless steel.

2.9.4 Forced circulation evaporator

Forced circulation evaporators are used if boiling point of the product on the heating surfaces is to be avoided as a result of the fouling characteristics of the products, and/or to avoid crystallization. The flow of the velocity in the tubes must be high, and high-capacity pumps are needed. The circulating liquid is heated while it flows through the heat exchanger and then partially evaporated when the pressure is reduced in the separator, cooling the liquid to the boiling point temperature corresponding to this pressure(Wiegand, 2009).

2.10 Single effect and multiple effect evaporators

Single-effect evaporator is used when the throughput is low, when a inexpensive supply of steam is available, when not cheap materials of construction must be used as is the case with corrosive feed stocks and when the vapour is so polluted so that it cannot be reused. Single effect units may be operated in batch, semi-batch or continuous batch modes or continuously.

Multiple-effect evaporators are an evaporator system in which the vapor from one effect is used as the heating medium for the next effect boiling at a lower pressure. Such a system is named a multiple-effect evaporator, and is effective in reducing total steam utilization for an evaporation process.

In a forward feed, the weak solution enters to the first effect where it is concentrated by the raw steam. The solution then flows onto the next effect where it is heated up by steam coming from the vapor space of the first effect. The solution is then concentrated further ahead of it flows to the third effect and so on.

On the other hand, the backward feed system, the solution is fed to the final effect and flows to the reverse direction with the raw steam being fed to the first effect that now holds the most concentrated solution. The raise in latent heat with decreasing pressure and additional radiation losses however results in the steam economy (defined as mass of vapor produced per unit mass of steam consumed) being increasingly lower as the number of effect is increased(Guahati, 2013, Macedonia, 2014).

2.10.1 Factors affecting the design and operation of an evaporator

The wide dissimilarity in liquor characteristics takes the process of an evaporator from simple heat transfer to a separate art. Some of the majority important properties of evaporating liquids are as follows (Ermis,et al, 2017, Rughurman, 2011).

1. Concentration

Dilute solutions may usually have liquid characteristics close to that of water, but as the quantity of solids increases, the solution becomes more and more distinctive. The density and viscosity rises with the increase in solid content until the solution saturated or too sluggish. In such cases, solids will deposit and of the solvent is progressively evaporated and it will require constant removal of solids to avoid clogging of pipes. Boiling point of solution may also rise with increase of solid contents and the boiling temperature of this solution may differ considerably from that of water at the same pressure.

2. Foaming

Solutions that tend to foam during evaporation need to be handled carefully. Formation of stable foam may cause the liquid to be carried over with the vapor leading to high entrainment losses.

3. Temperature Sensitivity

Certain solutions such as those of pharmaceutical and food products are sensitive or can be damaged at high temperatures. Evaporation of solvent from such heat sensitive materials would involve that their exposure to high heat be as minimum as possible, with evaporation occurring in the minimum amount of time. Specifically designed evaporator is needed in such cases.

4. Scale

Several solutions deposit scale on heat transfer surfaces cause to rapidly diminishing values of overall heat transfer coefficients. These should be cleaned from the tubes by shutting off the evaporators. If the scale is hard, in the cleaning will have difficulty and rise its expensiveness. Some evaporators are designed to avoid scale formation or are designed so that the scales are removed without difficulty.

5. Materials of Construction

Evaporators are generally made out of steel but certain solutions may damage ferrous materials or contaminate them. Hence other materials for example copper, nickel, stainless steel, aluminum, lead etc. can be used. Since these materials are not cheap, high heat transfer rates become especially desirable to reduce the first cost of the equipment. Many other liquids characteristic may be considered also, for example specific heat, heat of concentration, freezing point, and gas liberation on heating, toxicity, explosion hazards, radioactivity and the necessity for sterile operation. Because of the variation in liquid characteristics several different evaporators have been designed. The choice of the design depends primarily on the characteristics of the liquid.

2.10.2 Choosing the optimum number of effects for a caustic recovery plant

The cost of each effect of an evaporator per square foot of surface is a function of its total area and decreases with area, approaching an asymptote for very large installations. Thus an investment required for a number of effects is equal to the number of effects times the cost of one effect. The optimum number of effects should be determined from an economical balance between the savings in steam obtained by rising the number of effects and the added investment required(Shah & Bhagchandani, 2012).

For a caustic recovery plant, apart from the water evaporation rate, a textile mill's whole energy usage is reported to be the most important factor in selecting a suitable evaporation plant, as the number of effects, pre-heaters and additional heaters determines how much hot water is required. After the evaporation process, heat can be used to generate hot water. However, extra steam will not be needed when the evaporation plant operates within the mill's hot-water requirements. To achieve this, the evaporation plant must be designed accordingly. To have the correct number of effects, a total cost calculation is needed, which should take into account the mill's total heat usage and the actual evaporation plant itself. The necessary investment costs must be set against the savings for the mill's total energy requirements, fresh lye, the reduced quantity of waste liquid and the expenditure for neutralizing agents.

With a rising number of effects, the specifically required heating steam (kg/h heating steam per kg/h water evaporation) is reduced. Investment costs, however, increase with the rising number of effects; therefore it is usually not recommended to have more than four effects because are not economical. A water evaporation rate of 5,000 kg/h is believed to demand a two- or three-effect plant, with a water evaporation rate of 15,000 kg/h requiring a three- to four-effect plant (Zohra**,** 2011).

2.11 Optimization

Optimization applications during the mercerization steps are another option for textiles to maximize the benefits from the process. It is more economical and beneficial for the final treatment and to reduce the cost of mercerization

General processes that can be optimized can be given as follows;

• Chemical system evaluations

- Solution preparation system evaluations

- Water pre-treatment studies

• Auxiliary process optimizations

- Heat exchanger temperature optimizations
- Hot water/steam system analysis

• Product flow optimizations

- Process simplifications
- Energy conservation audits
- Thermal efficiency of process piping and vessels
- Water reuse studies and recycles

2.12 Mathematical calculations for evaporation

The quantitative balance for the evaporation process is divided into two parts: (Shah and Bhagchandani, 2012, Ermis et al., 2017)

- Mass balance
- Energy flow balance

The mass balance is given by the mass conservation law:

$$m_{in} = m_{ex} \quad \text{.....(2-1)}$$

m_{in} : Fed in mass

m_{ex} : Drawn off mass

In the case of a rising film evaporator, the balance is as follows:

$$m_{feed} = m_{conc} + m_{cond} \quad \text{.....(2-2)}$$

The quantity of heating steam consumed can be used to calculate the average concentrating and condensing capacity

$$p_{conc} = \frac{m_{conc}}{m_{HSC}} = [kg/kg] \quad \text{.....(2-3)}$$

$$p_{cond} = \frac{m_{cond}}{m_{HSC}} = [kg/kg] \quad \text{.....(2-4)}$$

The evaporative capacity of an evaporator relates to the quantity of heating steam required to evaporate 1kg of condensate. In this case, the reciprocal of the condensating capacity is equal to the evaporative capacity.

$$p_{evap} = \frac{1}{p_{cond}} = \frac{m_{HSC}}{m_{cond}} = [{}^{kg}\!/_{kg}] \quad \text{..... (2-5)}$$

The energy flow balance records and calculates all energy flows that are present in the system.

The basic requirement is that the sum of all energy flows is equal to zero.

$$\sum E_X = 0 \quad \text{..... (2-6)}$$

Consequently, all incoming energy flows into the system E_{in} in are positive and all outgoing energy flows E_{ex} are negative.

$$E_{in} - E_{ex} = 0 \quad \text{..... (2-7)}$$

The incoming energy flow E_{in} includes only the incoming flow of heating steam into the evaporator. The energy flow of the heating steam is calculated from the mass flow rate of the heating steam and the specific enthalpy of the steam state:

$$E_{HS} = \bar{m}_{HS} * h_{HS}(P, T) \quad \text{..... (2-8)}$$

The outgoing energy flows E_{ex} include the following:

- Heating steam condensate flow
- Concentrate flow
- Cooling water flow
- Condensate flow
- Heat loss flow

The energy flow for the heating steam condensate flow is calculated from the mass flow rate and the enthalpy as follows:

$$E_{HSC} = \bar{m}_{HSC} * h_{HSC}(p, T) \quad \text{..... (2-9)}$$

For the concentrate flow, the energy flow is calculated using the following equation:

$$E_{conc} = \bar{m}_{conc} * h_{conc}(P, T) \quad \text{.....(2-10)}$$

The energy flow for the condensate flow is calculated in the same way as for the concentrate flow:

$$E_{cond} = \bar{m}_{cond} * h(p,T) \quad \text{..... (2-11)}$$

For condensation and the cooling of the condensate, the energy flow is calculated from the cooling water flow of the condenser and the temperature difference between the inlet and the outlet.

$$E_{cool} = \bar{m}_{cool} * cp(\Delta T) \quad \text{..... (2-12)}$$

The heat loss flow is given by rearranging the energy balance:

$$E_{loss} = E_{HS} - E_{HSC} - E_{conc} - E_{cond} - E_{cool} \quad \text{..... (2-13)}$$

In addition to the incoming and outgoing energy flows, there is also a constant energy flow inside the rising film evaporator from the shell into the inner evaporator tube. To calculate this energy flow, the following assumptions are made:

- Only the condensation heat from the heating steam is transferred into the inner tube and the condensate exits the shell at boiling point.
- The transfer does not result in any losses. Based on these assumptions, the energy flow inside the evaporator is calculated using the following equation:

$$E_{evap} = E_{HS} * (h'' - h') \quad \text{..... (2-14)}$$

Here, h′′ stands for the specific enthalpy of the steam and h′ stands for the specific enthalpy of the heating steam condensate.

The basic individual efficiencies can be calculated from the individual energy flows using the following equation.

$$\eta_x = \frac{E_x}{E_{HS}} \quad \text{..... (2-15)}$$

2.13 Caustic soda recovery from mercerization process

2.13.1 Caustic Soda Recovery by membrane technology

The principle for the different separation systems using membrane is basically depends on the pore size of the membrane and the size of the particle that is to be separated. The membrane categories are based on size and molecular weight of particles which can be separated from the entering stream the pressure-driven solid-liquid separation processes in MF, UF, NF and RO(Guo.Zhou, and Lv, 2013).

Membrane technology developed in the current years has likewise found its application in caustic soda recovery. A commercial product called SelRo membrane is claimed to be capable of recovery and concentrating of NaOH and acids in industrial processes(Prado, et al and Marangoni, 2015) . Another commercial product called Alkasave marketed by the same membrane producing company is also successful in reclaiming strong alkaline cleaners from cleaning-in-place (CIP) streams in dairies and beverage plants (Hufemia, 2013). It consists of small membrane plants with tubular alkali-stable membranes that are attached to the main NaOH tank. In the recovery system, caustic waste is collected in a feed vessel and filled to the membrane unit. The membrane constantly removes dissolved and suspended organic contaminants from the caustic solution and the purified caustic soda obtained in permeate can be sent back to the main NaOH tank. To maintain the flux, water is added to the recovery system feed vessel in a diafiltration process.

The recovery of caustic soda therefore results to the reduction of input concentrated caustic solution in the industry. Although membrane technology is a good this method is not practical because textile wastes (chemicals) are highly fouling forming wastes. it can't operated even for one hour without cleaning but possible with combined membranes such as micro filtration, ultra filtration, nano filtration/RO but this has also operational difficulty(kyung, 2016).

The other method is using evaporators. This method is the most practical method and foreign textiles industries are using widely. The use of multiple effect evaporators are good enough according to the studies discussed above in the literature review. Evaporators are energy intensive (80-90^{0}c) but this heated waste (after recovery) can be used as heating steam (there is no energy waste for evaporators)(Sharma & Sharma, 2015). So this paper will use evaporator for recovery of caustic soda from mercerization process in textile industry.

2.14 Previous works

This section discusses about the various works carried out in the past on a laboratory scale as well as the successful installments on an industrial scale whereby economical recovery of caustic soda by evaporation has been achieved.

2.14.1 Laboratory work:

Attempts to set up a pilot plant whereby 4% caustic soda solution can be concentrated to 20% solution have been made in the past times. Two such latest works are discussed below:

Rahman and Khan tried to designing an evaporation unit that used a steam as a heating medium, for evaporating water at a rate of approximately 20 kg/hr. An average feed flow rate of 26 kg/hr of 4% solution was used to a circulating line coming from the bottom of a flash chamber. The solution was passed through a heat exchanger that used the steam to heat up to a boiling point. The solution then entered a flash chamber where the vapor disengaged, increasing the concentration of the solution to an average 20%. The vapor generated from the flash chamber was used for heating the feed solution.

The design provided an evaporation rate of 5.7 kg/hr per square foot of heat transfer surface, with an overall heat transfer coefficient that ranges from 1700 to 2500 $W/m^2.K$, whereby the 4% feed solution was ultimately concentrated to a 15 to 23% of caustic soda. The process was found to be operable economically feasible when installed in textile industries with an appropriate scale up. Their equipment was found to evaporate 0.8 kg of water for every kilogram of 20 psig steam consumed.

Shifa and Nayeema designed an experiment which uses flue gases from a burner to heat up the weak lye solution in a horizontal evaporator. The flue gas was passed through the tube side while the weak lye solution is in the shell side of the evaporator system. The vapor generated from the evaporation was used for heating (as Preheater) the feed solution before it entered to the evaporator. Shifa and Nayeema also reported a laboratory experiment whereby they found that the foaming characteristic of the weak lye solution required that the shell accommodating the hot solution by keeping only half filled, to allow enough gaps for vapor separation and the foam formed during the operation.

The investigators reported they have evaporated approximately 19 kg/hr of water to produce a product solution concentration of 20 to 21% and an average feed flow rate of 18 kg/hr of 4% solution. The evaporation rate was found to be 4.74 kg /hr with an overall heat transfer coefficient of 78.72 W/m^2 K and with consumption of natural gas 3.52 Nm^3/hr.

2.14.2 Industrial work

Recovery of weak lye from textile wastes has been carried out since 1900 for both woven and non woven fabrics. The processes in general involve the use of multiple effect evaporators though recent technologies that make use of ultra filtration and nano filtration processes are emerging. Evaporation provides a full recovery of the caustic in the wastewater. Its main disadvantage however lies in the operational difficulties that arise from the use of many chemicals that are used during the processing. Such as, the use of surfactants in the processing leads to high degree of foaming in tanks and vessels during the recovery of the weak lye (Kyung, 2016). There are a number of suppliers of chemical recovery units they set up evaporation systems to recover caustic soda from effluents. They declare their setup to be successful with payback periods usually ranging from 2 to 3 years. Such companies include UNITOP Aquacare and Chemical Process Systems.

UNITOP Aquacare explains their process in the following manner: 40-50 gm/l weak caustic lye is collected from the impregnation and washing chamber of the mercerizing process unit and collected in a tank. This is taken to a filtration unit to filter the suspended solids, fluff and etc. The solution is then fed through a sequence of preheaters which preheats the wash liquor up to its boiling point using flash vapors, thus ensures minimum steam usage. The liquor in the tubes then will be in contact with the steam in the shell side of the first heater and starts to boil. The vapor liquor solution then enters a flash vessel through a lateral entry. The vapors then separated and enter the shell of the second effect. The boiling in the successive effect take place caused by lowering pressure created with the help of a water ring vacuum pump.

The concentrated solution enters the second heater because of the pressure difference and comes in indirect contact with the generated vapors in the first effect. The same system is repeated in every effect. The concentrated product is taken from the last effect by a product pump. This is taken to a storage tank. The weak lye is concentrated to 250-300 gm/l in case of dry mercerizer or 450-500 gm/l in case of wet mercerizer which can be reused with a minor topping up of caustic soda. Vapors formed at the last effect need to be condensed. This is done by the ‘hot water’ or an ‘adiabatic evaporator’ system. In the hot water system, water at room temperature is passed through tubes of an exchanger and it becomes hot due to the vapors. This hot water was not in contact and it is uncontaminated and thus can safely be used in the process or the boiler as per requirement. In some cases where the hot water formed cannot be effectively used the effluent is passed through the exchanger and sprayed in

an adiabatic Evaporator. There are various examples in literature of mills that have setup caustic recovery systems effectively. A case study was carried out on a textile industry which uses a multiple effect evaporation system for recovery and reuse of the weak lye from its mercerizing unit. This is discussed as below:

The Yangzhou Dyeing and Printing textile factory is found in the east of Yangzhou City. The mill is a medium sized plant that has annual production capacity of 70 million cubic meters of dyed and printed cotton and synthetic fabric. Like other printing and dyeing operations, the plant is very energy intensive mill. In this mill, concentrations of 320 gm/l of NaOH is used in the mercerizing range, with discharges of weak lye at 40-50 gm/l. Effluents from alkaline processing have a significant impact on water quality with pH levels at Yangzhou above 12.0 - 13.7. The three-effect process technology converts weak soda to concentrated soda through a multiple stage evaporation process with exterior heat used to evaporate weak soda lye recovered from discharge waters. Secondary steam is captured and used as heat to evaporate the remaining soda lye in reverse order through the third, second, and first evaporating chambers. After the evaporation stages have been completed, concentrated NaOH at 330 gm/l is available. The equipment costs of the caustic soda recovery facility was 3.89 million RMB yuan (6.5 RMB= 1 US$). In the previous before the setup the plant would purchase 7,760 tons of NaOH per year for fabric treatment. After the setup, the plant concentrates 4,820 tons of NaOH and consumes the balance of 2,940 tons from external sources. The project had a payback of three years period of (after construction is completed).

In Germany, Hannover, the company Körting Hannover uses a Caustic Soda Recovery System for treating the mercerizing lye whereby the weak lye separates into strong lye and vapor condensate. The heating medium used is steam. The strong lye, which is recovered, can then be reused and recycled for mercerizing unit. Körting explained during textile finishing processes, particularly the mercerization of cotton fabrics, of weak caustic lye (3 – 7 % NaOH) coming from the rinsing water leaves the stabilization part of the mercerizing process. The pollution depends on the condition of the materials during mercerization, affected by the method of pretreatment of the fabric. It is maintained that a singed, desized and well-washed material will not contaminate the lye too much, while bleaching and rinsing before mercerization is also thought to be a good method to retain a dirt free lye cycle according to Körting. With the Caustic Soda Recovery System requiring steam and cooling water, more or less the same amount of steam used for the recovery of the mercerizing lye is saved in the hot-water generation, where the cooling water is to be heated up to 60 – 80°C.

The company claims the system is very good energy efficient, especially when the hot-water generation is incorporated into the central hot water system, but there is will no direct contact between the heating steam and the weak lye, allowing the heating steam condensate to be re-used as boiler feed water without any additional treatment. In general according to Körting, it is better to generate a smaller amount of hot water at 85°C, for instance, than a larger amount at 60°C, which is then, requires to be heated up with direct steam just for the machines.

3. Materials and methods

3.1 Materials:

Materials and equipments required for experiment one (optimization of caustic soda concentration for wet mercerization of bleached cotton fabric)

- pH meter
- Tubantin Blue FF2GL 200 (direct dye)
- Bleached cotton fabric
- Gretag Macbeth color-eye 3100 spectrophotometer
- Measuring tanks and degree Baume

Materials and equipments required for experiment; evaluations of caustic soda recovery using evaporation

1. Rising film evaporator**:**

The experiment was used a rising film evaporator of length of 305mm , width of 100mm and a height of 1470mm. it has also a temperature measuring connections to measure an evaporator temperature.

L x W X H: Approx.305x100x1470 mm

Evaporator area: 0.082 m²

2. Cyclone separator

L x W X H: Approx. 255x110x550 mm

Material: Borosilicate glass

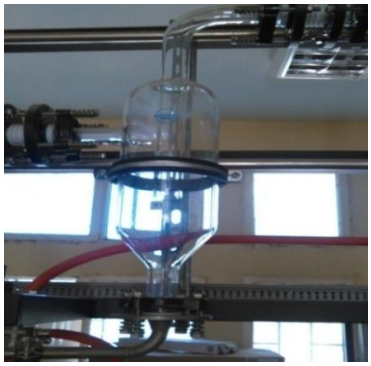


Figure 3-1 Cyclone separators

3. Condenser

L x W X H: Approx. 180x120x640 mm

Heat exchanger area: 0.2 m²

Material: Borosilicate glass

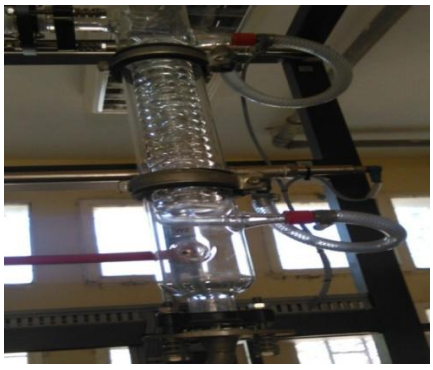


Figure 3-2 Condenser

4. Feed vessel

L x W X H: Approx. 410x310x340 mm

Capacity: Approx. 35 l

Figure 3-3 Feed vessel

5. Concentrate/ Condensate tank

Diameter x H: Approx. 300x280 mm

Capacity: Approx. 10.4 l

Glass tube: Borosilicate glass

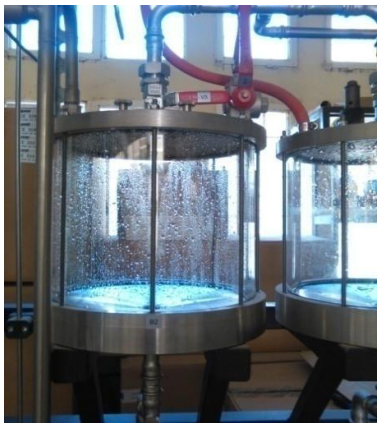


Figure 3-4 Concentrating/condensating tank

6. Measuring tanks (10L, 5L and 3L glass tanks)

7. Steam generator (with two boilers and 4-5 bar pressure)

8. Density areometer (Range 0 to 3 g/cm^3, temperature 0 to 40^{0}c)

9. Degree Baume (Range 0 to 100 ^{0}Baume, temperature 0 to 80 ^{0}c and sample volume minimum of 2ml)

10. pH meter (Range -2 to 16, accuracy +0.02 or -0.02, operating temperature 0 to 45 ^{0}c)

11. Vacuum Pump (L x W X H: Approx. 410x155x275 mm, intake volume at 50 Hz 90 L/min, Vacuum oil volume: 240 ml)

12. CIP pumps (L x W X H: Approx. 260x120x160 mm, Max. capacity 17 L/min)

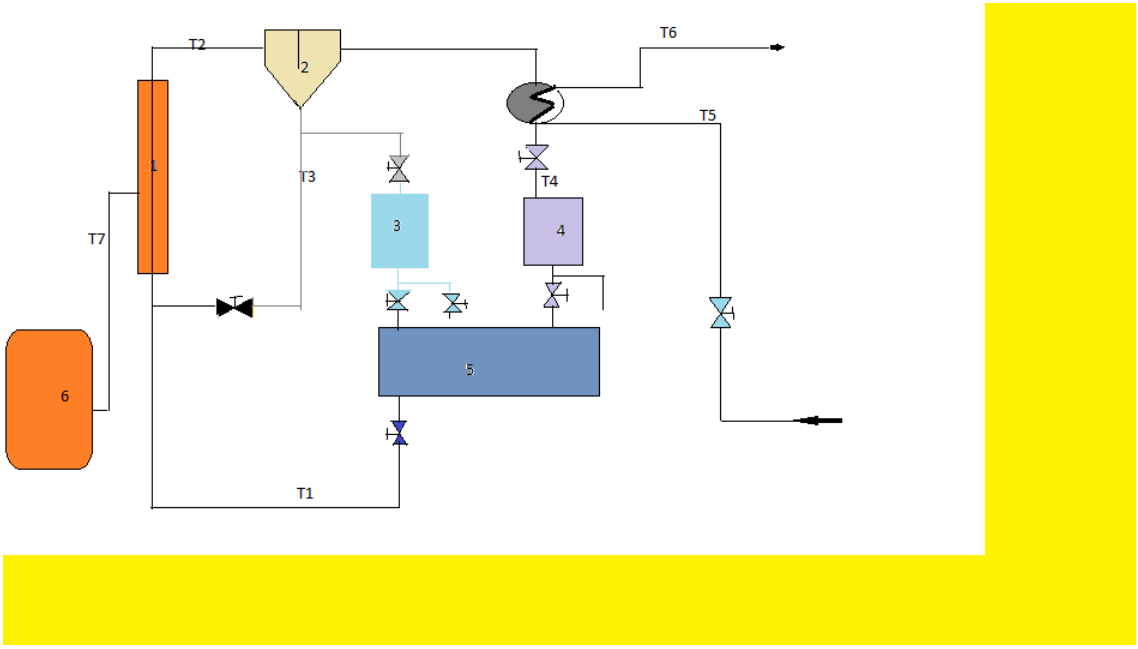


Figure 3-5 Process Flow diagram

1. Rising film evaporator
2. Separator
3. Concentrating tank
4. Condensating tank
5. Feed vessel
6. Steam generator

T1 feed temperature
T2 vapor temperature
T3 return temperature
T4 condensate temperature
T5 cooling water inlet temperature
T6 cooling water return temperature
T7 steam temperature

3.2 Experimental method for the evaluation of caustic soda recovery

The description of the process is shown as Figure 3.5 mentioned below. A 5% by weight caustic soda solution is to keep in the feed tank. This solution is sent to the tube side of rising film evaporator and the heated solution was rise to the cyclone separator at the top side of the evaporator. The cyclone separator allows the separation of the liquid and vapour phases to be observed. Separation occurs due to the centrifugal forces resulting from the incoming steam in the cyclone. The vapour phase emerges at the head of the cyclone and is fed into the condenser. The concentrated liquid accumulates in the lower section of the cyclone and flows into the return pipe. The solution will pass vertically upward through the tubes while the hot steam coming from the steam generator into the shell side of the Evaporator. The solution inside the tubes took heat from the steam on the shell side and began to boil. In the cyclone separator, the boiling solution from the Evaporator flashed generating vapor and leaving a

concentrated caustic soda solution in the lower section of the cyclone separator. The vapor leaving the cyclone separator was Condensate by passing through the condenser.

The following operating parameters should be viewed as constant:
Heating steam pressure p1 = 1.1 bar abs.
Cooling water flow rate f_2 = 350 l/h.

After completing the experiment, the following values were determined:

Mass of concentrate
Mass of condensate
Mass of heating steam condensate

3.3 Mercerization effluents characterization

Experimental studies started with the characterization of the caustic wastewater that originates from mercerization processes. There are a number of different mercerizing lines (washing stages) in the plant. During the study, several samples were taken from Alameda textile caustic discharging mercerizing lines of the mill to characterize caustic mercerizing effluents.

Six samples were collected from different streams at the point of their discharge. These samples were collected from different sections like washing stage one, washing stage two and washing stage three. Textile wastewater samples were collected in polyethylene bottles at the outlet. The samples were analyzed for various physicochemical parameters like, TDS, pH, EC, BOD, COD and TSS.

3.4 Experimental method for the optimization of caustic soda concentration for mercerization of wet bleached cotton fabric

The caustic soda strength is expressed in % (w/v) meaning g/100 ml. Commercially bleached cotton fabric was treated with caustic soda solution of concentrations varying from 5-30% The percentage wet pick up was distinguished in each case. The padded fabric samples were rolled on glass rods, covered with plastic sheet and batched for 30 minutes. The padding and batching were done at room temperature (20-25°C). After the batching age the samples were frequently rinsed in cold water in anticipation of the samples showed neutral pH.

Dyeing**:** The cotton samples treated with varying concentrations of caustic soda including those used for mercerization were dyed with a direct dye in wet-on wet the samples after final

rinsing were squeezed. The dyeing were carried out at high temperature dyeing machine under following conditions

Tubantin Blue FF2GL 200 (Direct dye)

Sodium chloride……..10g/L

MLR ……1:30

Temperature……..80°C

Waiting time ……45 minutes.

The dyeing was commenced at room temperature. The temperature was raised to 80°C and dyeing continued at this temperature for 45 minutes. After dyeing the fabrics were rinsed three times with cold water, squeezed, and then pressed with hot iron and conditioned before K/S measurement.

The main center of attention of the optimization is limited to systematically set up the most appropriate choice of caustic soda concentration for mercerization for increased dye uptake. The main objective is to establish experimentally the most appropriate concentration of caustic soda to be used for mercerization.

Determination of color yield (K/S): The color yield or dye-uptake was determined by measurement of the K/S value of the colored samples on Gretag Macbeth color-eye 3100 spectrophotometer by following the standard procedure. The effect of mercerization on improvement of color yield is well known. Measurements of color in dyed fabrics are analyzed in various ways, but for the purpose of comparing color yield changes due to mercerization as well as mercerization most workers have used the Kubelka-Munk function K/S where K is an absorption coefficient and S is a scattering coefficient(Kubelka, 2008). The ratio K/S increases with increasing depth of shade. The measurement of K/S gives the comparative color yield or dye uptake and not the absolute quantity of dye present on dyed sample.

4. Results and discussions

This section contains the discussions and results of two different experiments; optimization of caustic soda concentration for mercerization of wet bleached cotton fabric and evaluation of caustic soda recovery from the mercerization effluent. It also included the characterizations of the mercerizing effluents and the recovered results.

4.1 Characterization of mercerizing effluents

The effluents characteristics need to be properly monitored for better environmental protection. All the textile mills had their effluents having temperatures between 36.0^{o}C to 45^{o}C given by table below which is higher than the set limit by the NEQS.

As can be seen from Table 4.1 as we move along the post-washing stages of the mercerization line, pollution load in the wastewater decreases and nearly 80 % of the total load from finishing is discharged from the first two rinsing tanks. More importantly, more than 85 % of NaOH discharged from mercerizing process is in the wastewater from the first three stages of washing (water consumption and hence wastewater discharge from each post-washing tank are equal in volume). Based on this observation, it was thought that the wastewater mixture from the first three rinsing tanks would be tried to be treated for the alkali recovery instead of using the whole system effluent, considering the possible cost of the full-scale NaOH recovery system to be installed in the mill. It was also considered that this would be better in terms of performance of the recovery system too. Thus all pretreatment and treatment studies were run with the wastewater mixture originating from the first three post-washing tanks of the finishing mill.

Table 4-1 : Characterization of mercerization effluent

Parameters	NEQS	Run-6	Run -5	Run -4	Run -3	Run -2	Run -1
pH Value	6 -8.5	7.5	8.6	9.5	10.4	10.6	11.3
Temperature	< 40^{o} C	36	35.6	40.1	37.3	45.0	43.6
TSS	< 200 mg/l	934	1875	1619	1236	1159	1050
TDS	< 3500 mg/l	2469	5408	6481	4868	4025	3687
EC	400 mS/cm	195.3	182.4	175.7	96.3	20.1	1.5
BOD	< 80 mg/l	117.8	172.4	194	256.5	264.6	360
COD	< 150 mg/l	115.66	212.16	331.61	251.25	342.21	342.24

4.2 Optimization of caustic soda concentration for the mercerization of cotton

One of the problems considerable in textile industries is in mercerization process there is no very well defined limit of concentration of caustic soda for use. As from the literatures cited below the process of mercerization very well documented but contrary the information on mercerization is not satisfactory because of a wide range in recommending caustic soda concentration for mercerization (19-30% w/w),(Hersh, Carolina, & Mark, 2006) (20-30%w/w), (Gemci, 2010) and (19-26% w/w),(Tomasino, 1992)

4.2.1 Caustic soda concentration on color yield

Wet-on-wet dyeing: Commercially bleached cotton fabric samples were treated with varying concentrations of caustic soda (5-30% w/v) and then wet-on-wet (without drying) dyed with the direct dye as explained in the experimental section. The results are given below in table form.

$K/S = (1-R)^2/2R$ ……The relationship between K/S and spectrophotometer

Table 4-2 Effect of caustic soda (5-15%) on increasing K/S

Replication	Caustic soda concentration (%)							
	0%		5%		10%		15%	
	R	K/S	R	K/S	R	K/S	R	K/S
1	27.0079	12.5225	26.3546	12.1963	29.7914	13.9125	38.1011	18.0641
2	28.2596	13.1475	26.7856	12.4115	26.9382	12.4877	37.3134	17.6701
3	28.377	13.2063	26.3388	12.1884	27.1716	12.6042	35.9567	16.9924

Table 4-3 Effect of caustic soda (20 – 30%) on increasing K/S

Replication	Caustic soda concentration (%)							
	0%		20%		25%		30%	
	R	K/S	R	K/S	R	K/S	R	K/S
1	27.0079	12.5225	42.4472	20.2354	42.9521	20.4877	45.9944	21.0081
2	28.2596	13.1475	44.4817	21.2521	42.9521	20.4877	46.3291	21.3872
3	28.377	13.2063	42.9625	22.4929	45.5804	22.0341	45.8472	20.8731

For the authenticity of the results the experiments were designed on the basis of single factor completely randomized design using Design Expert Software. The sample without treatment with caustic soda (0% caustic soda) was used as control. The % increase in K/S values

indicated the increase in color yield on samples treated with varying concentrations of caustic soda compared to control (0% caustic soda). The trials were carried out with three replications and it has good consistency of the results. The completely randomized experimental design data for 21 experiments is given in below.

Table 4-4 complete randomized experimental design

Run No	Caustic soda (%)	K/S	%increase K/S
1	25	29.4877	69.6071
2	20	20.2354	61.5923
3	30	20.8731	66.0484
4	30	21.3872	74.7815
5	30	21.0033	67.7153
6	5	12.1963	-2.605
7	10	13.9125	9.99
8	25	22.934	77.9562
9	10	12.4877	-0.2786
10	5	12.4115	-5.598
11	0	13.2063	0
12	20	21.2521	69.7113
13	20	22.4929	79.6463
14	5	12.1884	-5.354
15	10	12.6042	0.2813
16	15	18.08641	44.4312
17	0	12.5225	0
18	0	13.1475	0
19	15	17.6701	41.1086
20	25	20.9877	68.6071
21	15	16.9924	35.695

The consistency of data for K/S was confirmed from normal probability plot of residuals, plotted by design expert software. As shown in figure 4-4, the points are fairly close to a straight line indicating the validity the experimental data. Residuals mean difference in the observed value (experimental value) and the predicted value or fitted value (by the software). In order to check that the residuals are normally distributed (in other words to check the validity of experimental data), the mostly used technique is to plot normal probability plot of residuals. If the residuals fall approximately along a straight line, the residuals are considered to be normally distributed. In contrast, if the residuals do not fall fairly close to a straight line, the residuals are not normally distributed and hence the data do not come from a normal population i.e. the data is not valid.

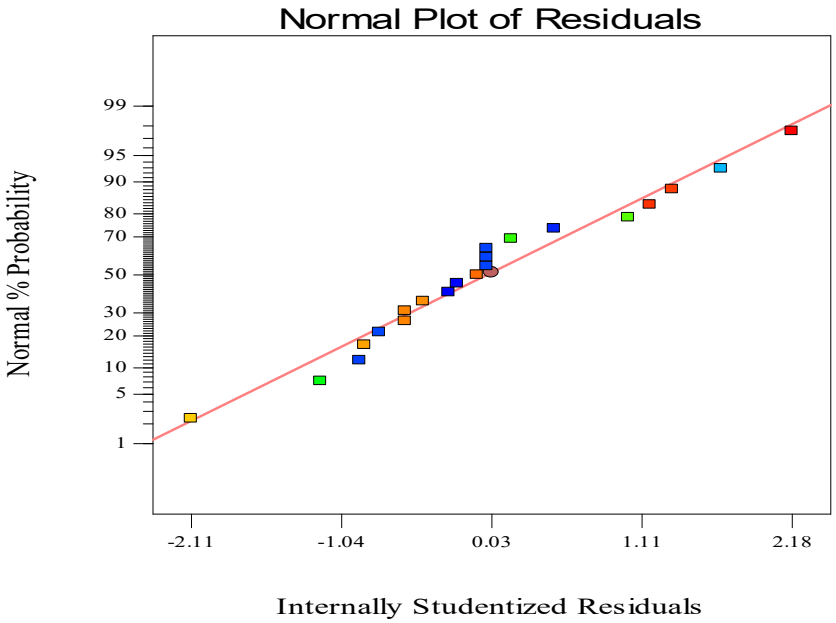


Figure 4-1Normal plot of residuals of K/S

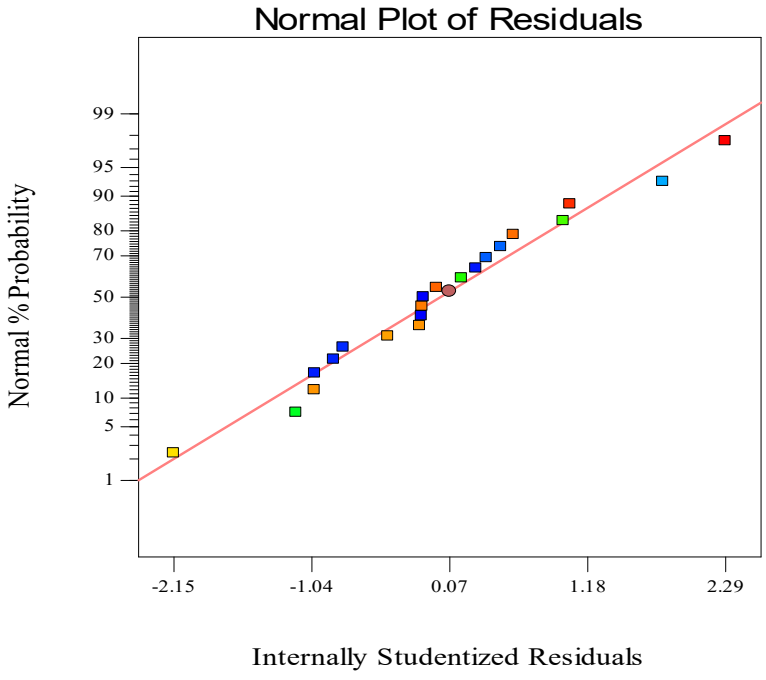


Figure 4-2 Normal plots of residuals of %increasing in K/S

The results of effect of caustic soda concentration on color yield for wet-on-wet dyeing measured as K/S for three replications are shown in figure.

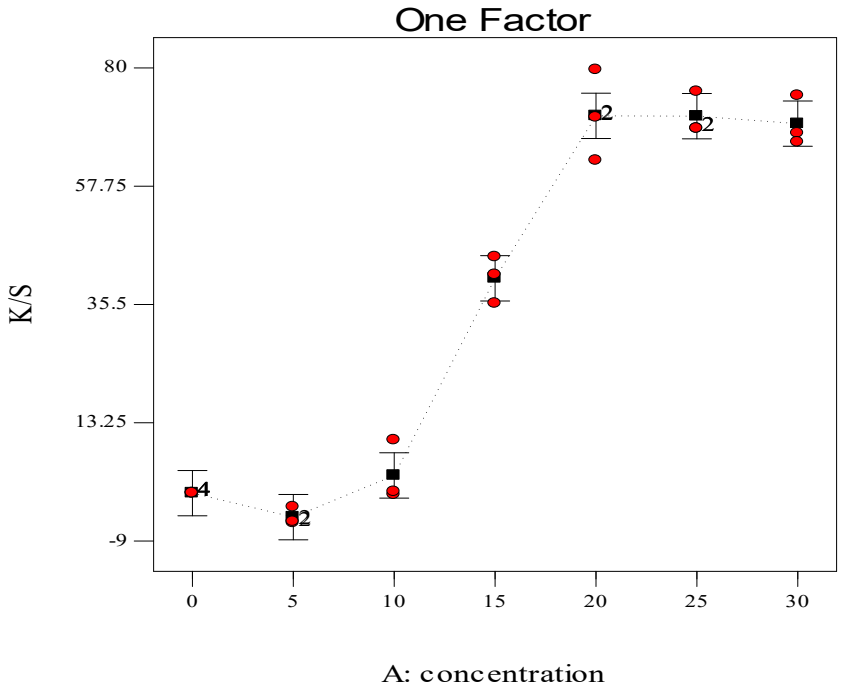


Figure 4-3 Effect of caustic soda on K/S

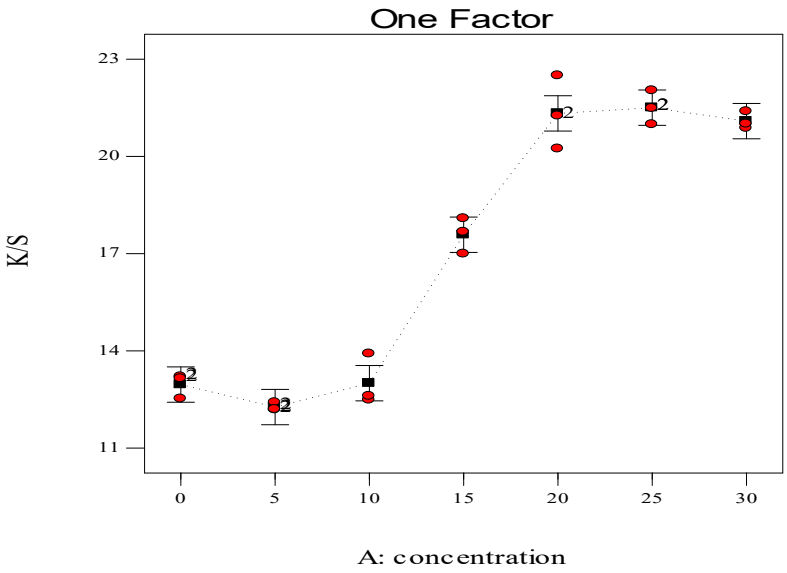


Figure 4-4 Effect of caustic soda on %increasing on K/S

The small circular points indicate the individual values where as the square points indicate the averages of the three individual points at that particular concentration of caustic soda. The broken line is drawn in such a manner that it connects the average points at each

concentration of caustic soda used for treatment. It can be observed that the effect of caustic soda concentration on dye uptake can be divided into four different stages.

Stage 1: from 0-5% marginal decrease in color yield.

Stage 2: from 5-10% slow increase in color yield.

Stage 3: from 10-20% rapid increase in color yield.

Stage 4: from 20-30% leveling off color yield.

The results can be interpreted on the basis of hydration of sodium hydroxide at different concentrations and its effect on swelling. There is some evidence that the degree of hydration of alkali hydroxide ions affects their ability to enter and swell cellulose fibers (M.Lee,2002). At low concentrations of sodium hydroxide, the diameters of the hydrated ions are too large for easy penetration into the fibers. As the concentration of caustic soda increases, the number of water molecules available for the formation of hydrates decreases and therefore their size decreases. Small hydrates can diffuse into the high order, or crystalline regions, as well as into the pores and low-order regions of cellulose (Elsevir, 2000, Hersh et al.2006).

The results of the effect of caustic soda concentrations on color yield can be interpreted on the basis of the above facts. Up to 5% caustic soda concentration effect on color yield is marginally decreased indicating that there may not be further breaking of hydrogen bonds of already water swollen fiber due to high degree of hydration at low concentration caustic soda. From 5%-10% caustic soda concentration, the fiber swelling tends to increase as indicated by the slow increase in color yield. From 10%-20% caustic soda concentration, the increase in color yield can be related to the high rate of swelling along with possibility of hydrogen bond breaking in the inter crystalline regions. Further than 20% caustic soda concentration, there may be excessive swelling resulting in the jamming of fiber structure with the result, the increase in dye uptake is leveled off. Therefore from the experiment result obtained, the optimum concentration for mercerization of bleached cotton fabric was 20-23%.

4.3 Caustic soda recovery evaluations

The experiment was conducted with changed parameters, the evaporator pressure, heating steam supply pressure and feed flow rate.

The table below shows the experimental results with variations of the evaporator pressure, heating steam supply pressure and feed flow rate.

Table 4-5 experimental results with variations of the parameters

Run#	P_3	P_2	F_1	t(s)	m_{conc}	m_{cond}	m_{HSC}
1	6	-0.3	400	2405	516	3108	1191
2	6	0.00	350	2412	1134	2670	1244
3	4.5	-0.3	350	2402	412	3893	1256
4	4.5	0.00	300	2401	1012	2991	1131
5	4.5	-0.6	400	2434	242	4290	1592
6	4.5	-0.3	350	2402	412	3893	1256
7	3	-0.3	300	2406	403	1500	1295
8	4.5	-0.3	350	2402	412	3893	1256
9	4.5	-0.6	300	2410	248	4277	1482
10	3	-0.3	400	2412	244	4295	1626
11	6	-0.3	300	2409	531	3093	1215
12	4.5	0.00	400	2403	946	1736	1314
13	4.5	-0.3	350	2451	412	3893	1256
14	3	0.00	350	2401	892	1798	1427
15	4.5	-0.3	350	2434	412	3893	1256
16	3	-0.6	350	2406	243	4286	1578
17	6	-0.6	350	2427	256	4068	1414

The table below shows the average temperatures recorded during the individual experiments. The temperatures recorded are based on one temperature measurement per experiment. They are average temperatures, which remain almost constant for the entire duration of the experiment.

Table 4-6 Average temperatures measured during the experiment

	T1[°C]	T2 [°C]	T3 [°C]	T4 [°C]	T5 [°C]	T6 [°C]	T7 [°C]
1	20.60	92.43	91.51	18.01	12.80	16.09	102.80
2	19.25	91.03	89.32	17.80	11.95	17.67	102.79
3	20.20	89.78	88.90	17.22	12.60	17.83	102.66
4	19.85	85.27	84.30	15.01	11.63	19.94	102.75
5	19.35	81.75	80.85	16.25	12.45	24.01	102.77
6	16.4	82.45	82.36	18.01	12.43	17.65	102.63
7	16.7	82.56	82.47	17.60	11.98	18.02	102.44
8	16.8	82.89	82.59	17.54	12.32	19.45	102.51
9	19.3	88.34	88.02	17.01	12.67	20.79	102.53
10	20.1	92.32	92.08	17.25	12.54	24.65	102.03
11	19.2	91.34	91.01	18.03	11.87	23.07	102.93
12	19.0	90.86	90.80	18.00	11.97	24.03	102.64
13	18.5	89.78	89.72	17.73	13.01	22.12	102.68
14	15.9	83.42	83.29	17.26	12.92	17.58	102.43
15	16.6	85.60	85.49	17.54	12.39	18.06	102.69
16	17.0	86.01	85.92	17.60	11.87	23.77	102.41
17	18.4	88.94	88.21	17.02	13.05	25.06	103.01

Evaluation of the experiment can be divided into determination of the mass balance, subsequent determination of the concentrating capacity and determination of the energy flow balance. The recovery of caustic soda using rising film evaporator; the produced caustic soda concentration increases with increasing heating steam supply pressure and vacuum pressure as shown in the table 4-7 below. Based on this, the maximum concentrating capacity of the solution was noted for 6 bars; heating steam pressure and 0 bar of evaporator pressure the concentrating capacity was 91%. The experiment with the highest capacity was selected for further process recovery procedure, in this case experiment 2 was selected which is optimum recovery attained.

Table 4-7 The determination of the mass balance and concentrating capacity:

Run #	P_3	P_2	F_1	t(s)	m_{conc}	m_{cond}	$m_{cond}+m_{conc}$	m_{HSC}	rate of evaporation(kg/hr)	Pconc
1	6	-0.3	36	2405	516	3108	3624	1191	4.65	0.43
2	6	0	19	2412	1134	2670	3804	1244	3.98	0.91
3	4.5	-0.3	19	2402	412	3893	4305	1256	5.83	0.32
4	4.5	0	2	2401	1012	2991	4003	1131	4.48	0.89
5	4.5	-0.6	36	2434	242	4290	4532	1592	6.34	0.15
6	4.5	-0.3	19	2402	412	3893	4305	1256	5.83	0.32
7	3	-0.3	2	2406	403	1500	1903	1295	2.24	0.311
8	4.5	-0.3	19	2402	412	3893	4305	1256	5.8	0.32
9	4.5	-0.6	2	2410	248	4277	4525	1482	6.38	0.16
10	3	-0.3	36	2412	244	4295	4539	1626	6.41	0.15
11	6	-0.3	2	2409	531	3093	3624	1215	4.6	0.43
12	4.5	0	36	2403	946	1736	2682	1314	2.6	0.71
13	4.5	-0.3	19	2451	412	3893	4305	1256	5.71	0.32
14	3	0	19	2401	892	1798	2690	1427	2.69	0.62
15	4.5	-0.3	19	2434	412	3893	4305	1256	5.75	0.32
16	3	-0.6	19	2406	243	4286	4529	1578	6.41	0.15
17	6	-0.6	19	2427	256	4068	4324	1414	6.03	0.18

The complete randomized results are give blow from the Design expert® 7.0.0 software.

Table 4-8 Complete randomized experimental results from Design expert® 7.0.0 software

std	run#	factor 1 (p_3)	factor 2 (p_2)	factor 3 (F_1)	P conc
1	9	6	-0.3	36	0.433
2	1	6	0	19	0.912
3	2	4.5	-0.3	19	0.325
4	13	4.5	0	2	0.895
5	5	4.5	-0.6	36	0.15
6	3	4.5	-0.3	19	0.325
7	7	3	-0.3	2	0.311
8	4	4.5	-0.3	19	0.325
9	14	4.5	-0.6	2	0.168
10	12	3	-0.3	36	0.15
11	11	6	-0.3	2	0.433
12	8	4.5	0	36	0.72
13	17	4.5	-0.3	19	0.325
14	15	3	0	19	0.621
15	6	4.5	-0.3	19	0.325
16	16	3	-0.6	19	0.15
17	10	6	-0.6	19	0.181

Consequently as the concentrating capacity varies with the different parameters, this indicate us the evaporating rate also varies at different heating steam supply pressure (P_3), evaporator pressure (P_2) and feed flow rate (F_1). The Experimental results, can be analyzed using Design expert® 7.0.0 software.

From table 4-8 above, the maximum concentrating capacity 0.912(91.2%) was obtained at an experiment number 2 at 19 l/hr, of feed rate, 6 bar of heating steam supply pressure, and 0.0 bar evaporator pressure. While the minimum capacity 0.152(15.2%) was obtained at experiment number 5; (4.5 bar,-0.6bar and 36l/hr heating steam supply pressure, evaporator pressure and feed flow rate respectively) at experiment number 10, (3 bar,-0.3bar and 36l/hr heating steam supply pressure, evaporator pressure and feed flow rate respectively) and at experiment number 16, (3 bar,-0.6bar and 19l/hr heating steam supply pressure, evaporator pressure and feed flow rate respectively) The decrease and increase of the concentrating

capacity was depending on the level of factors. There resulting data, from the above table, were analyzed using Design expert® 7.0.0 software to determine the effect of evaporator pressure, heating steam supply pressure and feed flow rate. All experiments were carried out in a randomized order to minimize the effect of unexpected variability in the observed response due to irrelevant factors.

Consecutively to determine whether or not the quadratic model is significantly affected by the parameters listed in the design, it was fundamental to carry out analysis of variance (ANOVA), in the tables blow. The probability values (P-values) were used to perform as a device to check the significance of each coefficient, which also showed the interaction strength of each parameter. The smaller the p-values are, the bigger the significance of the corresponding coefficient.

Table 4-9 Design summary

Design Summary			
Study Type	Response Surface	Experiments	17
Initial Design	Box Behnken	Blocks	No Blocks
Design Model	Quadratic		

Response	Name	Units	Obs	minimum	Maximum	Trans	Model
Y1	concentrating capacity	%	17	0.15	0.912	None	Quadratic
Factor	Name	Units	Type	Low actual	High Actual	Low Coded	High Coded
A	heating steam supply (p_3)	Bar	Numeric	3.00	6	-1	1
B	evaporator pressure(p_1)	Bar	Numeric	-0.6	0	-1	1
C	feed flow rate	l/hr	Numeric	300	400	-1	1

Response: Concentrating capacity

ANOVA for Response Surface Quadratic Model

Table 4-10 Analysis of variance table [Partial sum of squares]

source	Sum of Square	DF	Mean Squares	F	Source Prob > F	
Model	0.98	9	0.11	341.28	< 0.0001	significant
A	0.065	1	0.065	204.67	< 0.0001	
B	0.78	1	0.78	2433.46	< 0.0001	
C	0.015	1	0.015	48.51	0.0002	
A^2	1.053E-004	1	1.053E-004	0.33	0.5838	
B^2	0.091	1	0.091	285.00	< 0.0001	
C^2	5.813E-004	1	5.813E-004	1.82	0.2192	
AB	0.017	1	0.017	54.58	0.0002	
AC	6.480E-003	1	6.480E-003	20.30	0.0028	
BC	6.320E-003	1	6.320E-003	19.80	0.0030	

Residual	2.24E-03	7	0.0003192
Lack of Fit	*2.24E-03*	3	*7.45E-04*
Pure Error	*0*	4	*0*
Cor Total	0.98	16	

From the above Table 4-11 analysis of variance F- Value is a test for comparing model variance with residual (error) variance. If the variances are close to each other, the ratio will be close to one and it is less likely that any factors have a significant effect on the response. The Model F-value of 341.28 implies the model is significant. Values of "Prob > F" less than 0.0500 indicate model terms are significant.In this case A, B, C, B^2, AB, AC, BC are significant model terms. A, B, C, B^2, AB, AC, BC are coded significant parameters. The "Lack of Fit F-value" of 0.000745 implies that, Lack of Fit is not significant relative to the pure error. Non-significant lack of fit is required since the model is wanted to be fitted.

Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. There is only a 0.01% chance that a Model F-Value this large could occur due to noise

Table 4-11 Model adequacy measures

Std. Dev.	0.018	R-Squared	0.9977
Mean	0.40	Adj R-Squared	0.9948
C.V.	4.50	Pred R-Squared	0.9636
PRESS	0.036	Adeq Precision	58.670

Coefficient of variation, the standard deviation expressed as a percentage of the mean; predicted Residual Error sum of squares, which is the a measure of how the model fits each point in the design; the R- squared measures the amount of Varian around the mean explained by the model; Adj R- squared is the measure of the amount of variation in new data explained by the model, and Adequate precision, this is a signal to disturbance ratio due to random error, the following table below, are used to decide whether the model can be used or not. The "Pred R-Squared" of 0.9636 is in reasonable agreement with the "Adj R-Squared" of 0.9948. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 58.670 indicates an adequate signal. Subsequently this model can be used to navigate the design space.

Table 4-12 Regression coefficients

	Coefficient		Standard	95% CI	95% CI	
Factor	Estimate	DF	Error	Low	High	VIF
Intercept	0.325	1	0.007991	0.306105	0.343895	
A-heating steam supply (p3)	0.090375	1	0.006317	0.075437	0.105313	1
B-evaporator pressure(p1)	0.311625	1	0.006317	0.296687	0.326563	1
C-feed flow rate	-0.044	1	0.006317	-0.05894	-0.02906	1
A2	-0.005	1	0.008708	-0.02559	0.01559	1.005882
B2	0.147	1	0.008708	0.12641	0.16759	1.005882
C2	0.01175	1	0.008708	-0.00884	0.03234	1.005882
AB	0.066	1	0.008934	0.044875	0.087125	1
AC	0.04025	1	0.008934	0.019125	0.061375	1
BC	-0.03975	1	0.008934	-0.06088	-0.01862	1

The regression coefficients and the corresponding 95% CI (Confidence Interval) High and Low were obtained in table below. If zero was in the range High and Low 95% Confidence Interval, the factors has no effect. From the 95% CI (confidence interval) High and Low values of each model term, a conclusion can be drown that the regression coefficients of the parameters and the interaction terms have highly significant effect in the caustic soda recovery process. Using the designed experimental data the quadratic polynomial model (soft ware) for the recovery of caustic soda from mercerization process; for the concentrating capacity of the rising film evaporator was retreated and given as an equation form below:

Final Equation in Terms of Coded Factors:

Concentrating capacity = $0.32 + 0.09A + 0.31B - 0.044C - 5.000E\text{-}003A^2 + 0.15B^2 + 0.012C^2 + 0.066AB + 0.040AC - 0.040BC$ ……..……..(4-1)

Final Equation in Terms of Actual Factors:

concentrating capacity = 2.27675 - 0.063583 * heating steam supply (p_3) +2.28625 * evaporator prussure(p_1) -7.38000E-003 * feed flow rate -2.22222E-003 * heating steam supply $(p_3)^2$ +1.63333 * evaporator prussure$(p_1)^2$ +4.70000E-006 * feed flow rate^2 +0.14667 * heating steam supply (p_3) * evaporator prussure(p_1) +5.36667E-004 * heating steam supply (p_3) * feed flow rate -2.65000E-003 * evaporator prussure(p_1) * feed flow rate ……..(4-2)

4.3.1 Diagnostic plot

Normal probability plot of the raw data used to check the assumption of normality. The consistency of data for concentrating capacity was confirmed from normal probability plot of residuals, plotted by design expert software. As shown in figure below, the points are quite close to a straight line indicating the validity of the experimental data. Residuals mean difference in the observed value (experimental value) and the predicted value or fitted value (by the software). In the analysis of variance, it is usually more effective (strait line) to do this with the residuals which is shown below. In visualizing the straight line, consign more highlighting on the central values of the plot than on the extremes.

For a valid model and satisfied assumptions, the residual value must be unstructured; in particular, they should be disparate to any other variable including the predicted response. A simple check is to plot the residuals versus the fitted (predicted) values. A plot of the residuals versus the rising predicted response values tests the assumption of constant variance.

Table 4-13 Diagnostics case statistics

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Student Residual	Cook's Distance	Outlier t	Run Order
1	0.15	0.13	0.023	0.75	2.574	1.988	10.340	16
2	0.18	0.18	1.25E-03	0.75	0.14	0.006	0.13	17
3	0.62	0.62	-1.25E-03	0.75	-0.14	0.006	-0.13	14
4	0.91	0.93	-0.023	0.75	-2.574	1.988	-10.340	2
5	0.31	0.33	-0.015	0.75	-1.637	0.804	-1.929	7
6	0.43	0.43	7.13E-03	0.75	0.798	0.191	0.774	11
7	0.15	0.16	-7.13E-03	0.75	-0.798	0.191	-0.774	10
8	0.43	0.42	0.015	0.75	1.637	0.804	1.929	1
9	0.17	0.18	-8.38E-03	0.75	-0.937	0.264	-0.928	9
10	0.9	0.88	0.016	0.75	1.777	0.947	2.221	4
11	0.15	0.17	-0.016	0.75	-1.777	0.947	-2.221	5
12	0.72	0.71	8.38E-03	0.75	0.937	0.264	0.928	12
13	0.33	0.32	0	0.2	0	0	0	6
14	0.33	0.32	0	0.2	0	0	0	8
15	0.33	0.32	0	0.2	0	0	0	15
16	0.33	0.32	0	0.2	0	0	0	13
17	0.33	0.32	0	0.2	0	0	0	3
* Case(s) with |Outlier T| > 3.50								

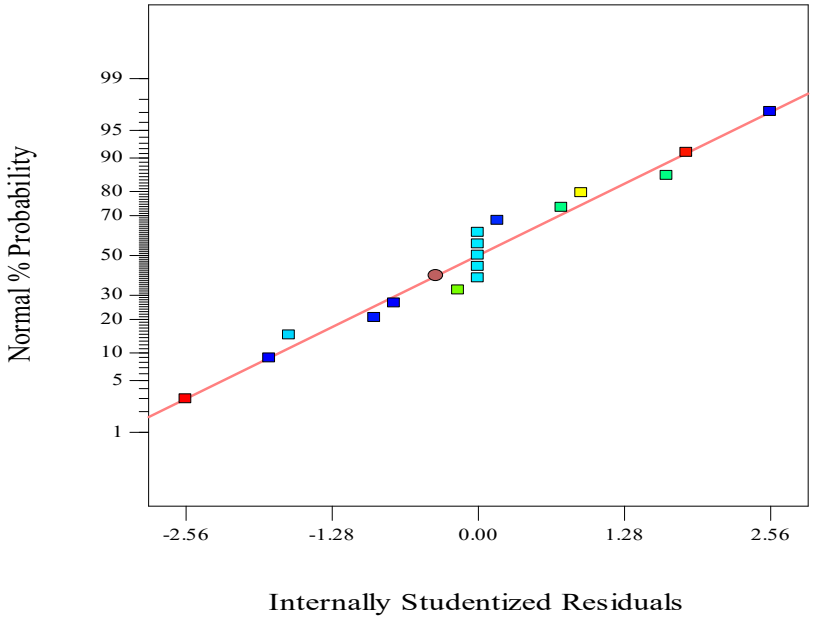


Figure 4-5 Normal plot of residuals

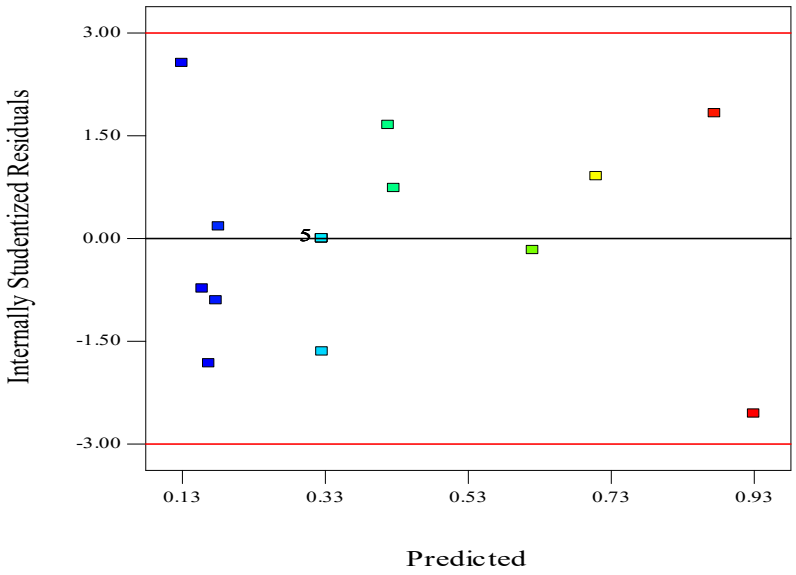


Figure 4-6 Plot of residuals versus model predicted values

Therefore from the above plot shows random scatter or unstructured which satisfied the assumption of the constant variance.

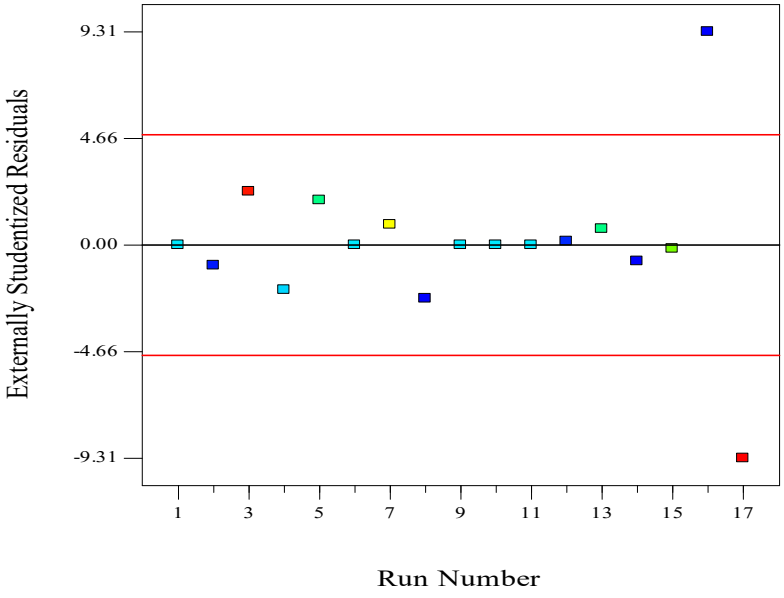


Figure 4-7 Externally studentized residuals

4.4 Individual Effects of experimental parameters (variables)

Parameters that can affect recovery of caustic soda indirectly they affect the concentrating capacity of the evaporator. The effects of the parameters on concentrating capacity are discussed below one by one. The best way of showing the effects of these parameters for the concentrating capacity are to generate response surface plots of the factors.

4.4.1 The effect of heating steam supply pressure

The resulting plot of heating steam supply pressure versus concentrating capacity, when feed flow rate and evaporator pressure were at actual factors, is plotted in the Figure below. As shown from the plot increasing heating steam supply pressure from 3 up to 6 bar, concentrating capacity increased from 22.96% to 41%, the reason is that increasing heating supply pressure means indirectly increasing the steam (heat) and this speed up the creation of vapour consequently the concentrating capacity increased.

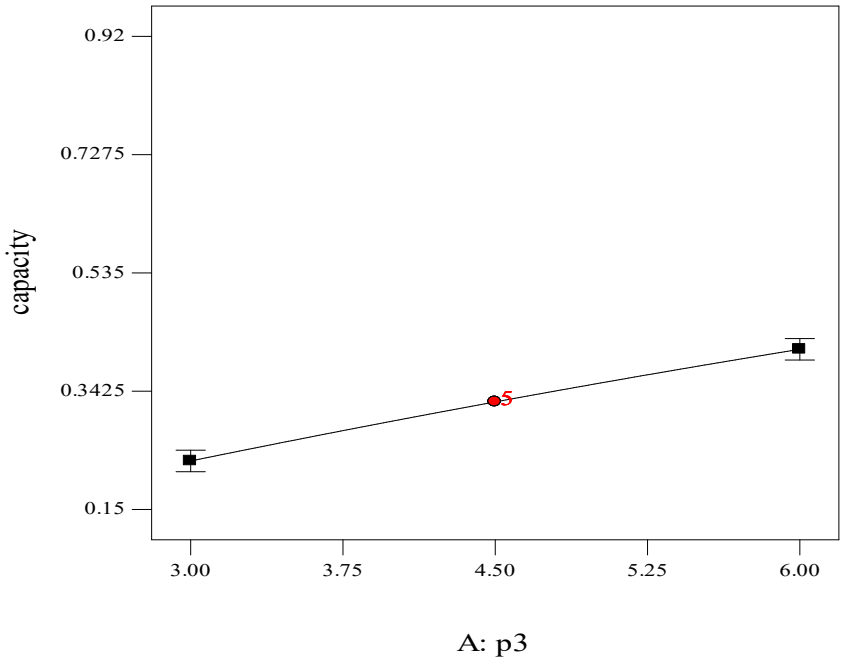


Figure 4-8 effect of heating steam supply pressure

4.4.2The effect of evaporator pressure (p_2)

The resulting plot of evaporator pressure versus the concentrating capacity, when the heating steam supply pressure and feed flow rate, is plotted in the Figure below. From the plot as evaporator pressure (p2) increases from -0.6 to 0 bar concentrating capacity also increased

slightly at the binging from-0.6 to -0.3 and rapidly increased as goes from -0.3 to 0.0bar. The concentrating capacity was increased from 16.01% to 78.3%. Therefore, evaporator pressure was found to be highly affects (from the other factors) the concentrating capacity of the evaporator; this due to the decreased value of boiling point of water at vacuum pressure.

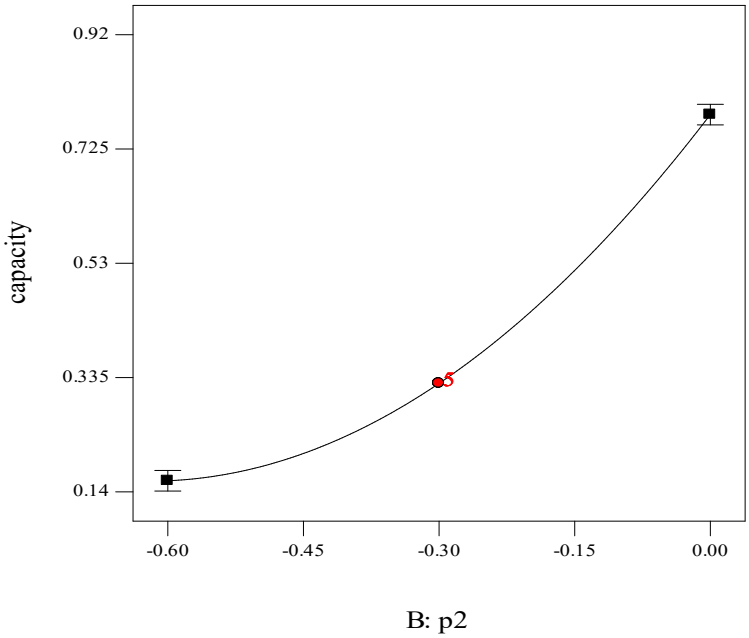


Figure 4-9 Effect of evaporator pressure

4.4.3 The effect of feed flow rate

The resulting plot of versus the concentrating capacity of the evaporator, when heating steam supply pressure and evaporator pressure were actual factors, is shown in the Figure below. As shown from the plot increasing from feed rate 2 to 36 l/hr, the concentrating capacity decreased from (38.07% to 29.27%) the reason was the increased amount of feed per small heat transfer area of the evaporator.

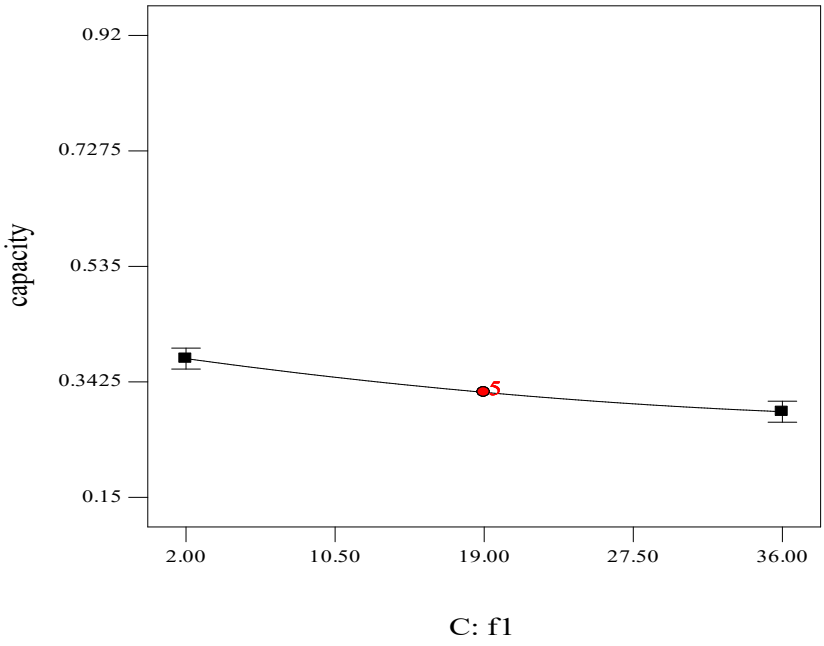


Figure 4-10 Effect of feed flow rate

4.5 Interaction effects of experimental parameters

The best way to show the effects of these parameters for recovery of caustic soda are to generate response surface plots of the equation. These effects are plotted in the following different figures as a function of the interactions of any two of the variables and the other value remains at actual value (at middle). For the interaction figures, black and red line indicates the two different parameters.

- The effects of heating steam pressure and evaporator pressure on the concentrating capacity when the feed rate was at the center point.

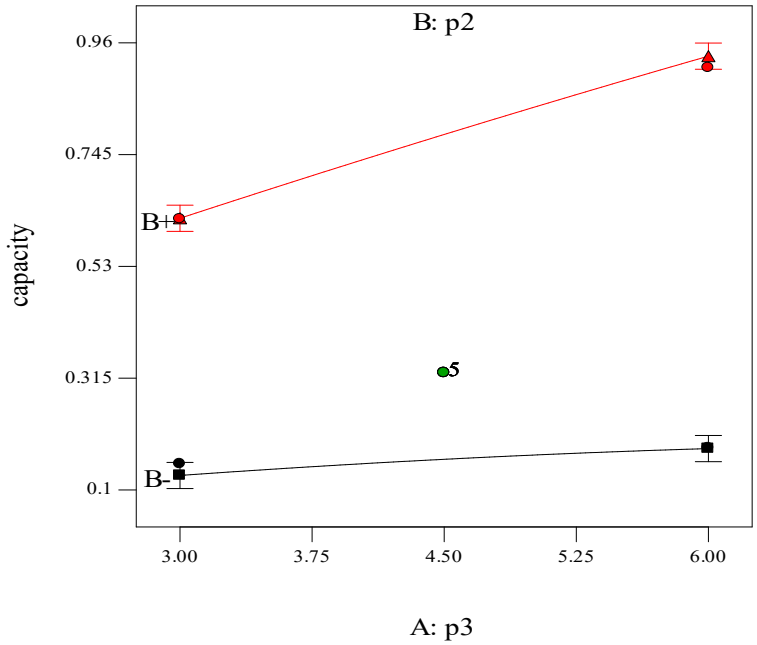
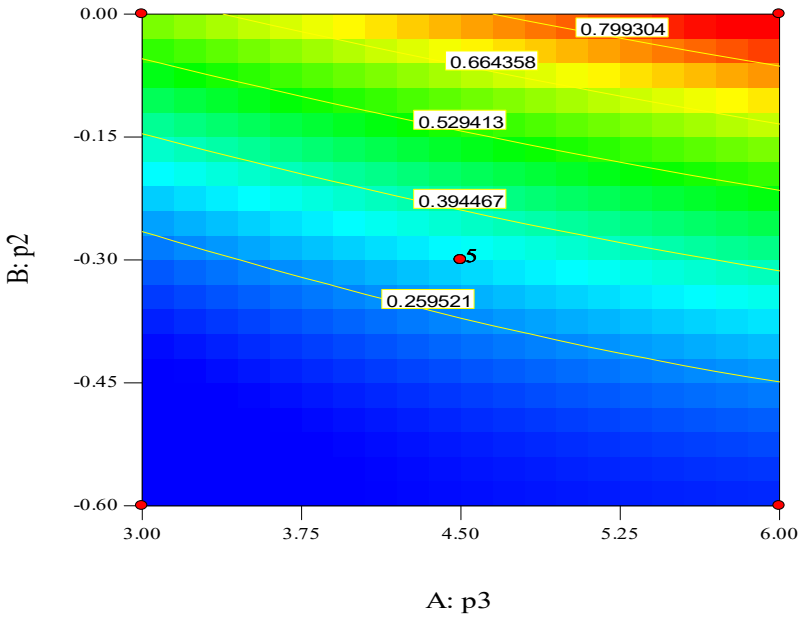


Figure 4-11 Contour plots of the effects of heating steam supply pressure and evaporator presure



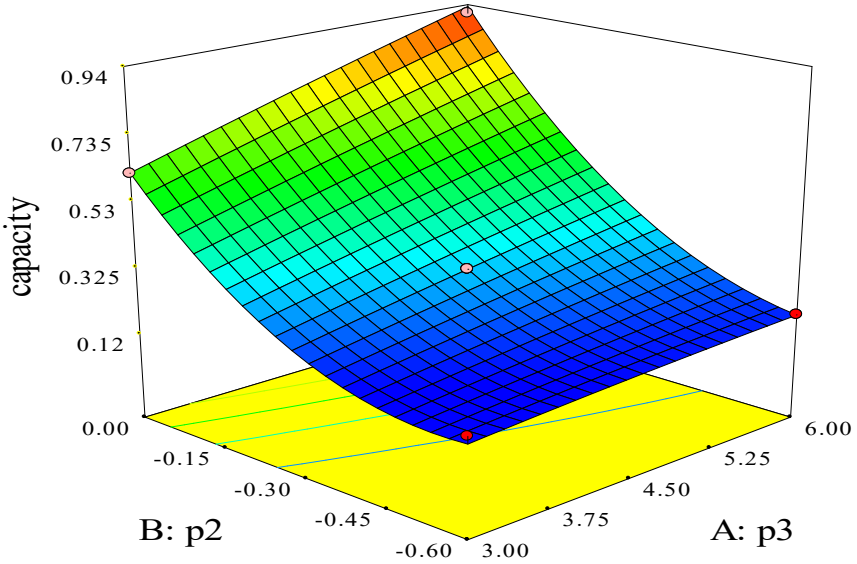


Figure 4-12 Response surface plots (3D) of the effects of heating steam supply pressure and evaporator pressure on concentrating capacity

The interaction effects of heating steam supply pressure and evaporator pressure when, feed flow rate was selected at the center point, are shown in figure 4-10. At the lower and higher levels of heating steam supply pressure and evaporator pressure, the concentrating capacity of the recovery level increased due to the increment of steam flow and lowered boiling point of water. Hence both heating steam supply pressure and evaporator pressure have strong relationship (interaction) on the concentrating capacity of the evaporator.

The contour plot graph shows the predicted response of concentrating capacity as a function of heating steam supply pressure and evaporator pressure was shown in figure 4-11. As heating steam supply pressure increases at lower level of evaporator pressure and as evaporator pressure increases at low level of heating steam supply pressure shows a positive effect on the concentrating capacity.

The response surface figure 4-12, obtained from heating steam supply pressure and evaporator pressure was slanted rectangular shape. Hence from the result, there were well defined optimums operating conditions.

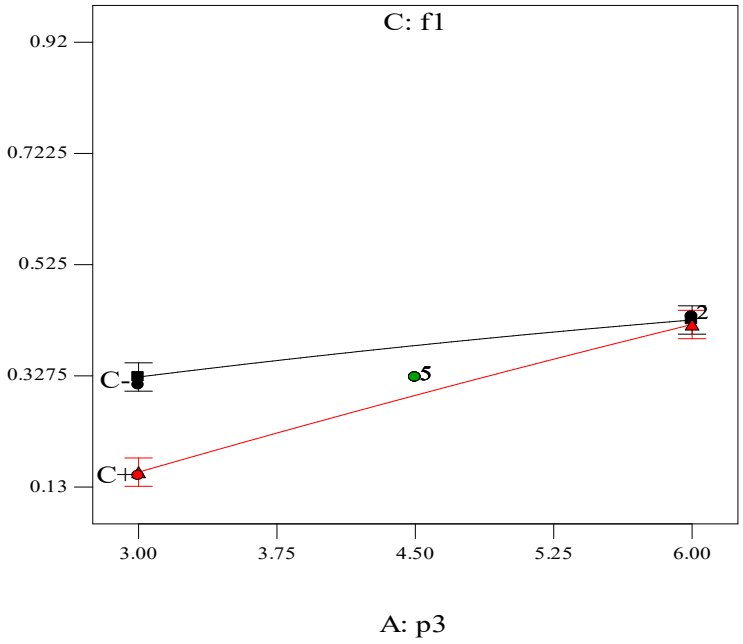


Figure 4-13 The effects of heating steam supply pressure and feed flow rate on the concentrating capacity, evaporator pressure was at the center.

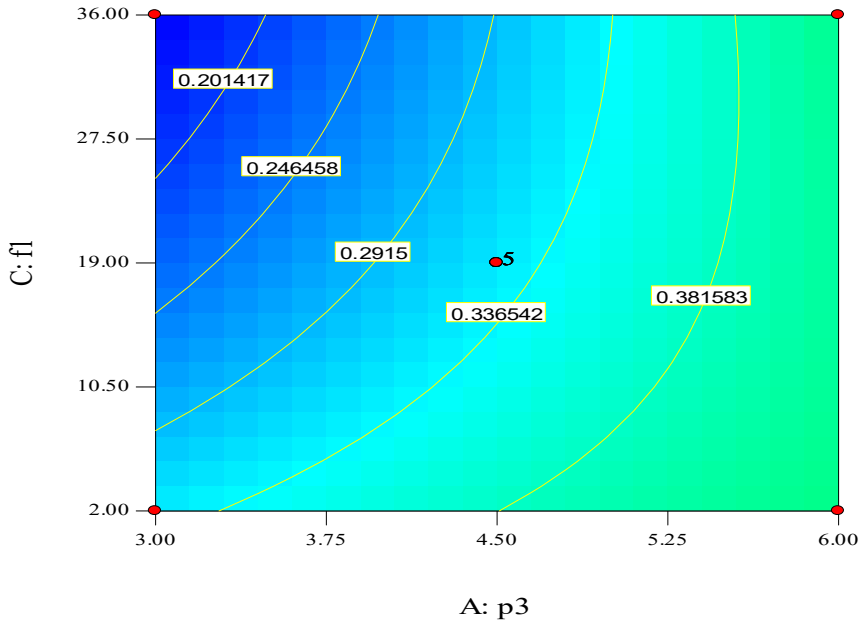


Figure 4-14 Contour plots of the effects heating steam supply pressure and feed flow rate on the concentrating capacity

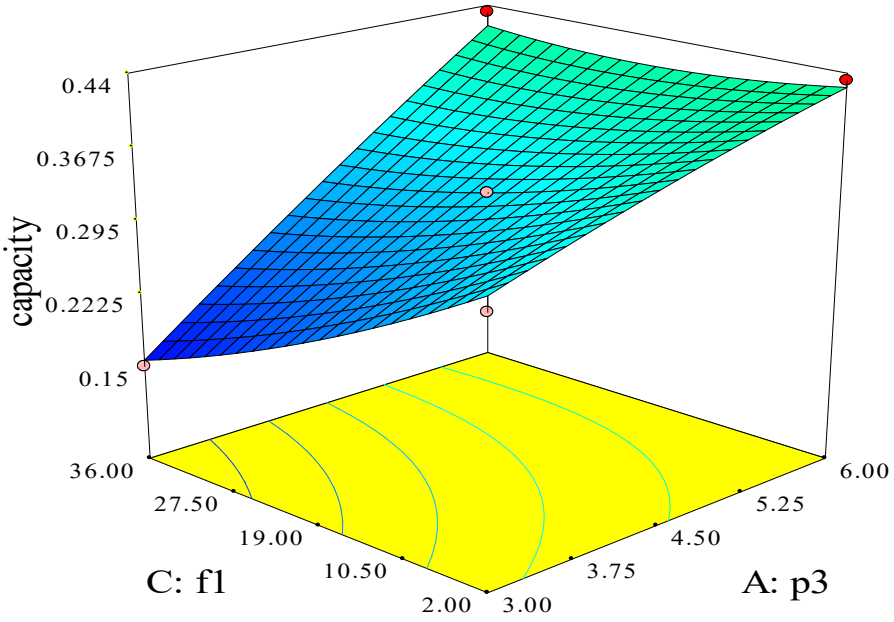


Figure 4-15 Response surface plots of the effects heating steam supply pressure and feed flow rate

For the interaction effects of heating steam supply pressure and feed flow rate when, evaporator pressure was at the center point, are shown in figure 4-13. As heating steam supply pressure increases concentrating capacity also increases. However, as shown from the graph at higher level of feed flow rate the concentrating capacity was decreased from lower level 31.1% to 15%. But due to the strong effect of heating steam supply pressure the interaction effect has positive effect on the concentrating capacity. From the contour plot graph showing predicted response of concentrating capacity as a function of heating steam supply pressure and feed flow rate was shown in figure 4-14.

The response surface figure 4-15, obtained from heating steam supply pressure and feed flow rate was slanted rectangular shape. It suggests that there were well-defined optimum operating conditions. This shows the heating steam supply pressure and feed flow rate can highly affect for concentrating capacity.

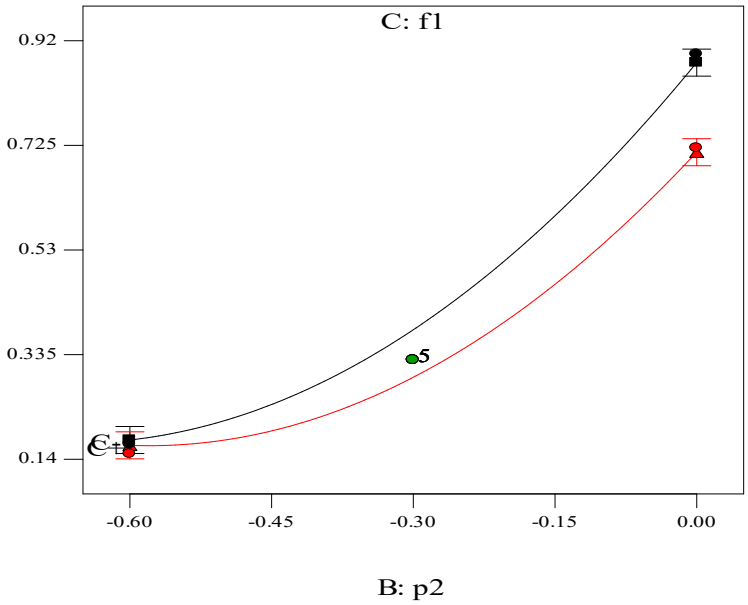


Figure 4-16 Interaction effect of evaporator pressure and feed flow rate on concentrating capacity

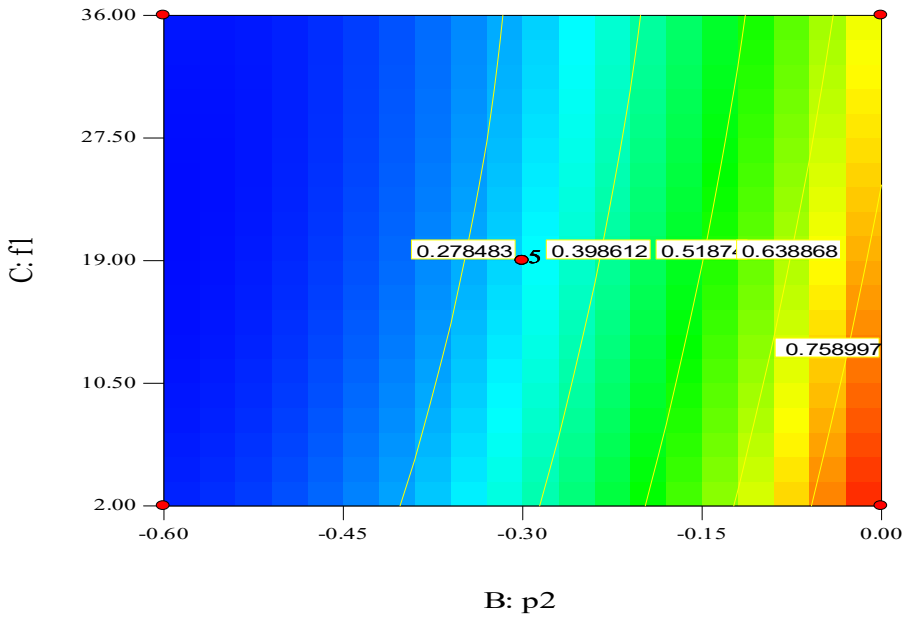


Figure 4-16 Contour plots of the effects evaporator pressure and feed flow rate on concentrating capacity

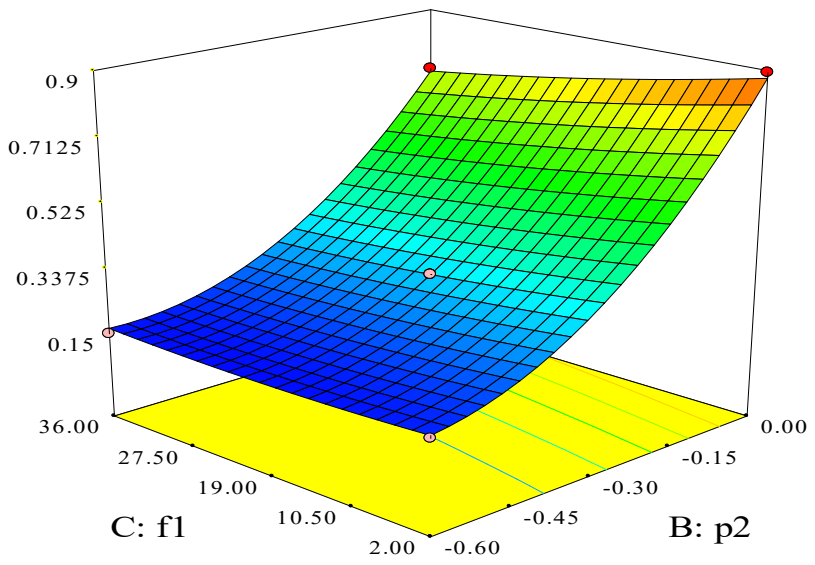


Figure 4-17 Response surface plots of the effects evaporator pressure and feed flow rate

For the interaction effects of evaporator pressure and feed flow rate are shown in figure, As evaporator pressure was at high level and the feed flow rate was at low level the concentrating capacity increased sharply where as the evaporator pressure was at high level and feed flow rate also at high level the concentrating capacity slightly increase relative to the evaporator pressure was at high level and the feed flow rate was at low level. Even though, the feed flow rate has a negative effect on the concentrating capacity due to insufficient heat surface area of the evaporator; interactively they have a positive effect on the concentrating capacity because of the strong effect of evaporator pressure. From the contour plot graph showing predicted response of concentrating capacity as a function of Evaporator pressure and feed flow rate was shown in figure 4-17.

The response surface figure 4-18, obtained evaporator pressure and feed flow rate was slanted rectangular shape. This suggests that there were well-defined best possible operating settings (conditions) and shows the evaporator pressure and feed flow rate has highly affected for the concentrating capacity.

As the parameters vary (P1, P2 and F1); it can be observed the effects of the parameters on the concentrating capacity shown in the figure below

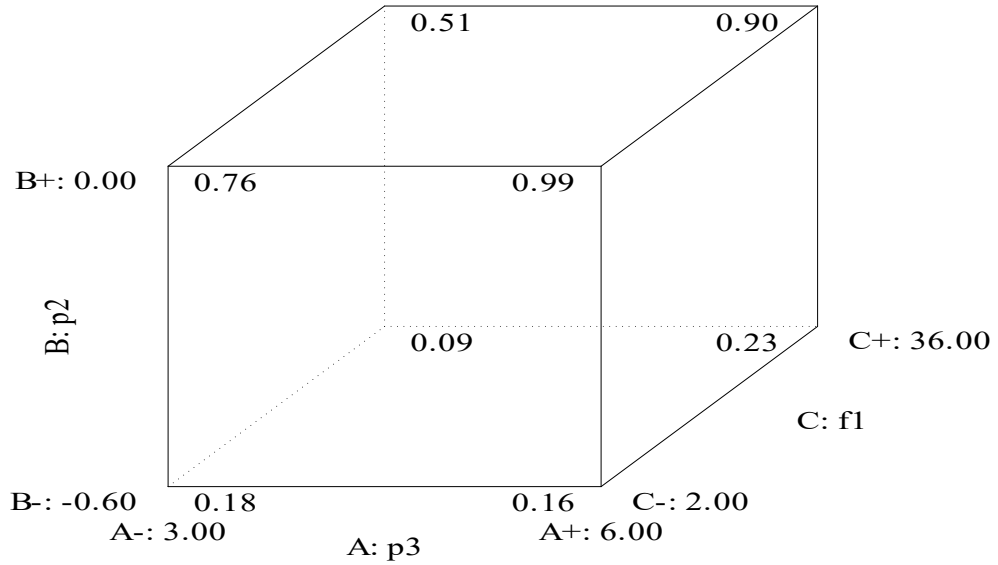


Figure 4-18 cube plot

All selected parametrs have an effect but heating steam supply pressure and evaporator pressure have very strong effect on the the disired out put. F_1(feed flow rate) has less effect relative to the other two parametrs.

It can be conculed that at high level of the two parametrs (P_2 and P^3) and at low level of feed flow rate (F_1) the concentrating capacity was at peak point (very high) with concentrating capacity of 99%. Contrarily , at lower level of P_2 and P_3 and high level F_1 the concentrating capacity is very low with 10%.

4.6 Process optimization

In the recovery of caustic soda, its capacity can be optimized by manipulating the parameters that has effects such as the feed flow rate, the heating steam supply pressure and the vacuum evaporator pressure. However, optimizing the response is difficult as the variation of each parameter with each other parameter is highly imprecise as we can observe from the previous sections. Consequently for that reason, in order to optimize the response (capacity), the function of desirability was applied using Design Expert software version 7.0.0 and numerical optimization was chosen. Numerical optimization presents a complete and advanced explanation of the most effective methods in optimization process. The upper and lower limit of each parameter (feed flow rate, the heating steam supply pressure and evaporator pressure) and its response as predicted by the model were provided based on the contour and surface plot obtained previously. The ultimate goal of this optimization was to obtain the maximum response that simultaneously satisfies all the variables at their desired properties. The table below shows constraints of each variable and the desired response.

Table 4-14 Optimazation constraints

Constraints Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight
P_3	maximize	3	6	1	1
P_2	is in range	-0.6	0	1	1
f_1	maximize	2	36	1	1
capacity	maximize	0.15	0.912	1	1

Table 4-15 solutions found for numerical optimization

Number	p_3	p_2	f_1	Capacity	Desirability	
1	3.18	0	36	0.53615	0.78053	Selected
2	3.16	0	36	0.53615	0.78045	-----
3	3.13	0	36	0.53615	0.78045	-----
4	3.24	0	36	0.53615	0.7804	-----
5	3.29	0	36	0.53615	0.78012	-----
6	3.22	0	36	0.53615	0.78	-----
7	3.34	0	36	0.53615	0.77962	------
8	3.39	0	36	0.53615	0.77906	------
9	3.43	0	36	0.53615	0.77842	-----
10	3.54	0	36	0.53615	0.77618	-----
11	3.18	0	34.49	0.53615	0.77443	-----
12	3	0	36	0.53615	0.77371	-----
13	3.17	0	33.02	0.53615	0.76806	-----
14	3	0	25.96	0.53615	0.73185	-----
15	4.44	0	23.15	0.53615	0.63621	-----

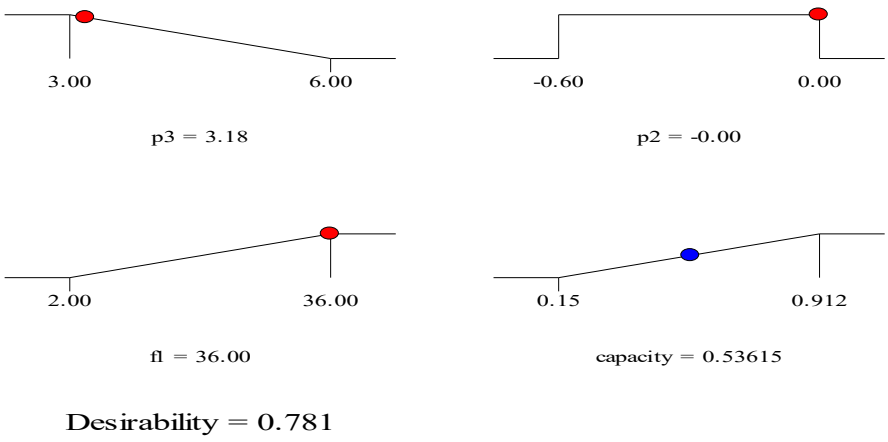


Figure 4-19 Ramp function graph

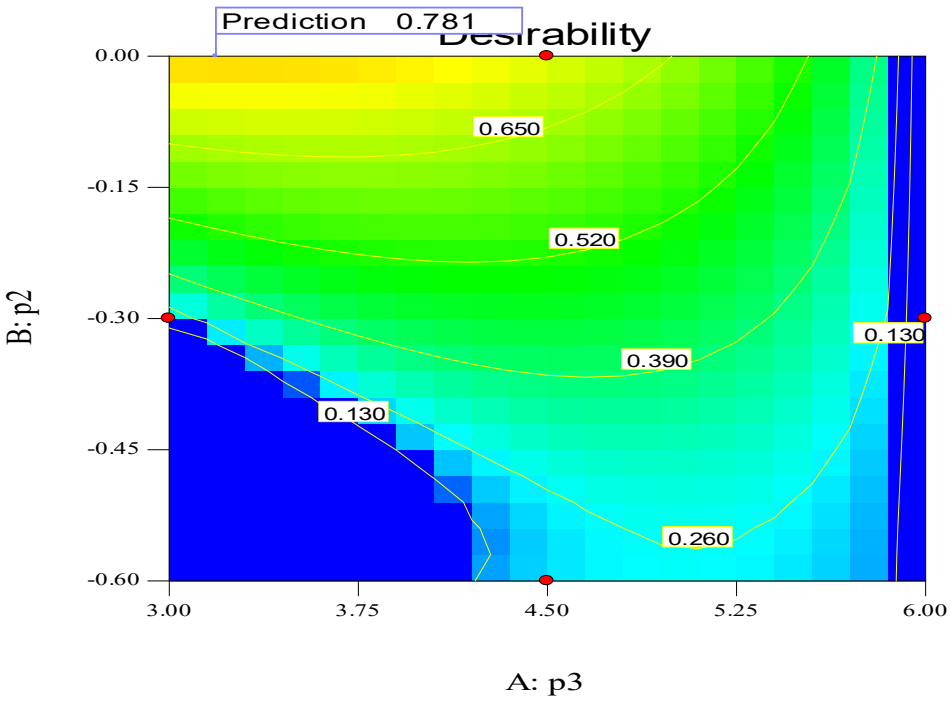


Figure 4-20 Contour plot of effects of heating steam pressure and feed flow rate on Desirability

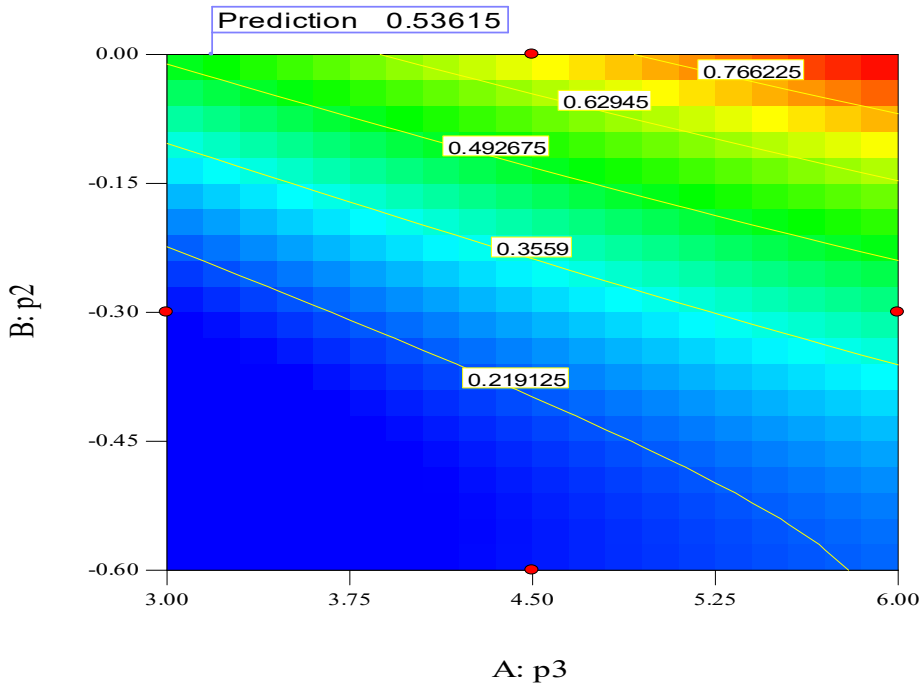


Figure 4-21 Contour plot of the predicted response as function of feed flow rate and heating steam supply pressure

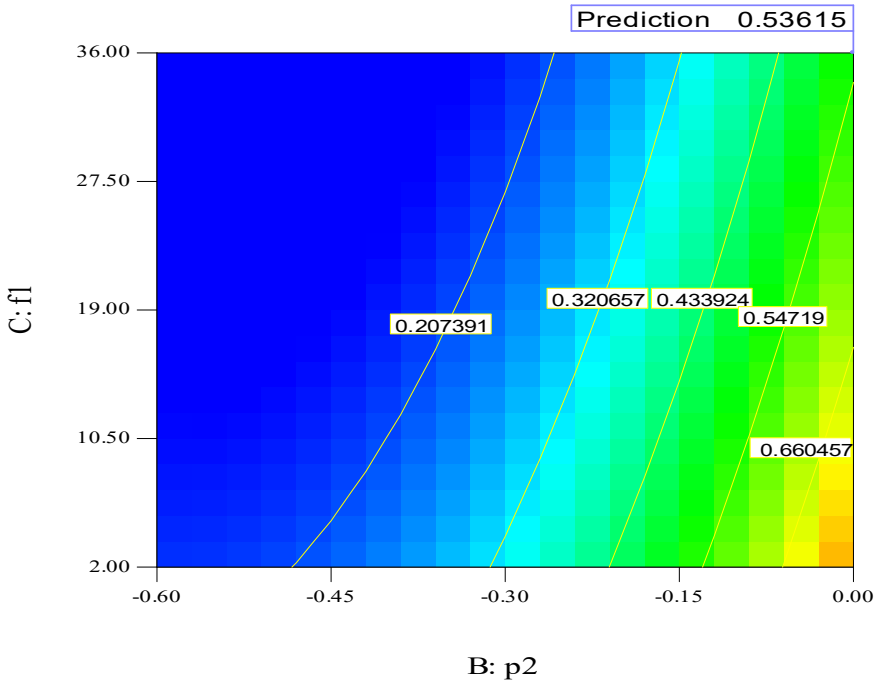


Figure 4-22 Contour plot on optimization as a function feed flow rate and evaporator pressure

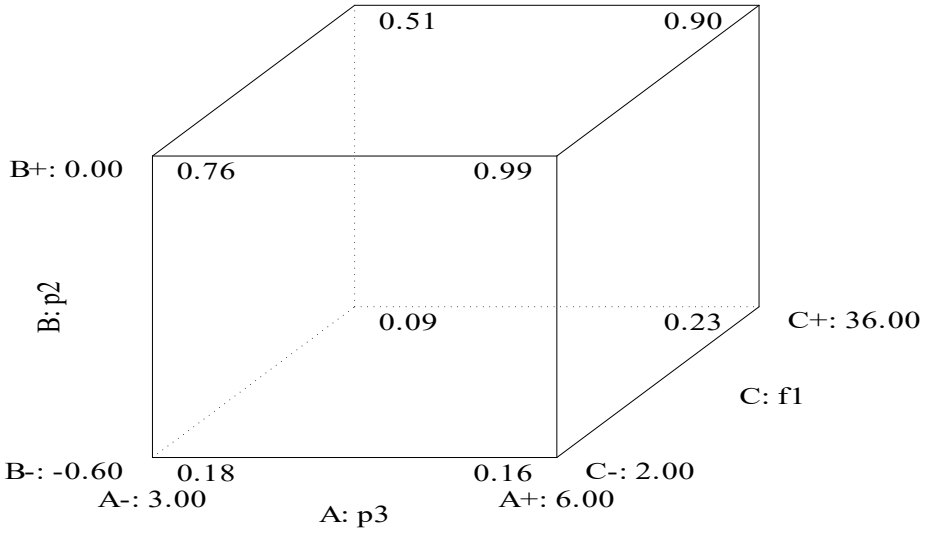


Figure 4-23 cube plots for the effects of parameters on desirability

Contour plot of the graph showing above in the optimization process predicted response of concentrating capacity as a function of heating steam supply pressure and acid feed flow rate was shown in figure (c). As heating steam supply pressure riches maximum value 3.18 bar at maximum level of feed flow rate 36 l/hr and as level of higher level of evaporator pressure shows a optimum concentrating capacity. The optimum response can be seen easily from a cube plot (e), obtained from heating steam supply pressure, feed flow rate and evaporator pressure was in no doubt shape to conclude the optimized parameters. Hence from the result, there were well defined optimums operating conditions. At optimum level evaporator pressure and at optimum level of heating steam supply pressure and the graph shows an optimum positive effect on the concentrating capacity. Therefore the optimized results of the process was given at an optimum value parameters heating steam supply pressure 3.18 bar, feed flow rate of 36 l/hr and evaporator pressure of 0.0 bar with optimum concentrating capacity of 53.62%.

4.6.1 Model validation

According to the central composite design result using Design-Expert® 7.0.0 software, an experiment with evaporator pressure, heating steam supply pressure and feed flow rate were conducted in order to investigate the effects of parameters in the design of the experiment. The experiment was carried out at the optimized conditions. Numerical optimization was carried out to maximize the response, using the response optimizer in Design expert®7.0.0. The optimal values test parameters were 3.18 bar of heating steam supply pressure, 0.0 bar evaporator pressure and 36 l/hr feed flow rate (obtained from Table above). Concentrating capacity of 53.62% (average) obtained and it was in good agreement with the predicted one. Therefore the model is considered to be good and reliable for predicting the concentrating capacity.

5. Conclusions and Recommendations

5.1 Conclusions

The characteristics results of the effluents from mercerization process does not abide by the national environmental quality standards and the effluents were alkaline in nature which should be properly monitored for better environmental protection. From the test of the chemical evaluation, the optimum concentration for wet mercerizing of bleached cotton fabric is 20 -23% and this reduces the unwanted expense for excess caustic soda.

According to the experiments conducted the selected parameters heating steam supply pressure and evaporator pressure have a positive effect on evaporation process but increasing feed flow rate have a negative effect on the evaporation process. The evaporation system has recovered the effluent with producing a caustic stream at reusable concentrations (20-23% NaOH). The process unit could evaporate water at a rate ranging between 2.24 kg/hr to 6.41 kg/hr with an approximate average of 5.72 kg/hr.

5.2 Recommendations

Some modifications can be made to the existing methodology for improving its operation such as further experiments can be conducted with a Preheater installed to the system of evaporation for a better evaporation rate in a short time and to save the steam generated for evaporation and a different evaporator with a greater heat transfer area can be used for a better investigation of the parameters

Further experimental investigations need to be carried out whereby the actual alkali effluent from a textile mill can be brought in and filtered and then used in the system to confirm the result and find other difficulties and to improve the quality of the recovered caustic soda and other also additional evaporator can be installed and the observation of values for a double effect evaporation system can be studied.

Since the effect of alkaline wastes on environment is very high, textile industries should adopt and exercise the caustic soda recovering system to save their costs for materials and energy and for a better and healthier environment.

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Appendix

A. Maximum allowed characteristics of waste water from textile industries

No.	Quality indicator	Units	MAC	Analysis method
1.	Temperature, T	^{0}C	40	-
2.	pH	pH units	6.5-8.5	SR ISO 10523-97
3.	Suspended solids (TSS)	mg/L	350	STAS 6953-81
4.	BOD	mg O2/L	300	STAS 6560-82
5.	COD	mg O2/L	500	SR ISO 6060/96
6.	Nitrogen (as |$NH4^+$1)	mg/L	30	STAS 8683-70
7.	Total phosphorus (as P)	mg/L	5.0	STAS 10064-75
8.	Total cyanides (CN)	mg/L	1.0	SR ISO 6703/1-98
9.	Sulphides and Hydrogen Sulphide (S2")	mg/L	1.0	SR ISO 10530-97
10.	Sulphites $(SO_3)^{2-}$	mg/L	2	STAS 7661-89
11.	Sulphates $(SO_4)^{2-}$	mg/L	600	STAS 8601-70
12.	Phenols (C_6H_5OH)	mg/L	30	STAS 7167-92
13.	Substances extractable with organic solvents	mg/L	30	SR 7587-96
14.	Biodegradable synthetic detergents	mg/L	25	SR ISO 7875/1.2-96
15.	Lead (Pb^2+)	mg/L	0.5	STAS 8637-79
16.	Cadmium (Cd^2+)	mg/L	0.3	SR ISO 5961/93
17.	Total Chromium (Cr^3+)+(Cr^6+)	mg/L	1.5	SR ISO 9174-98
18.	Hexavalent Chromium (Cr^)	mg/L	0.2	STAS 7884-91
19.	Copper (Cu^2+)	mg/L	0.2	STAS 7795-80
20.	Nickel (Ni^2+)	mg/L	1.0	STAS 7987-67
21.	Zinc (Zn^2+)	mg/L	1.0	STAS 8314-87
22.	Total Manganese (Mn)	mg/L	2.0	SR ISO 6333-96
23.	Free residual chloride (Cb)	mg/L	0.5	STAS 6364-78

source: (Savin & Butnaru, 2008)

B. Specific Gravity and Concentration of Caustic Soda Solution

d	Be	NaOH %	NaOH g / l	d	Be	NaOH %	NaOH g/ l
1.007	1	0.59	6.0	1.220	26	19.65	239.7
1.014	2	1.20	12.0	1.231	27	20.60	253.6
1.022	3	1.85	18.9	1.241	28	21.55	267.4
1.029	4	2.50	25.7	1.252	29	22.50	281.7
1.036	5	3.15	32.6	1.263	30	23.50	296.8
1.045	6	3.79	39.9	1.274	31	24.48	311.9
1.052	7	4.56	47.3	1.285	32	25.50	327.7
1.060	8	5.20	55.0	1.297	33	26.58	344.7
1.067	9	5.86	62.5	1.308	34	27.65	361.7
1.075	10	6.58	70.7	1.320	35	28.83	380.6
1.083	11	7.30	79.1	1.332	36	30.00	399.6
1.091	12	8.07	88.0	1.345	37	31.20	419.6
1.100	13	8.78	96.6	1.357	38	32.50	441.0
1.108	14	9.50	105.3	1.370	39	33.73	462.1
1.116	15	10.30	114.9	1.383	40	35.00	484.1
1.125	16	11.06	124.4	1.397	41	36.36	507.9
1.134	17	11.90	134.9	1.410	42	37.65	530.9
1.142	18	12.69	145.0	1.424	43	39.06	556.2
1.152	19	13.50	155.5	1.438	44	40.47	582.0
1.162	20	14.35	166.7	1.453	45	42.02	610.6
1.171	21	15.15	177.4	1.468	46	43.58	639.8
1.180	22	16.00	188.8	1.483	47	45.16	669.7
1.190	23	16.91	201.2	1.498	48	46.73	700.0
1.200	24	17.81	213.7	1.514	49	48.41	732.9
1.210	25	18.71	226.4	1.530	50	50.10	766.5

Source; (Safe Handling of Caustic Soda, 2006.)

C. Properties of Caustic Soda

Molecular formula: NaOH

Molecular weight: 40.00

Specific gravity: 1.48 (45% concentration)

1.50 (50% concentration)

Melting point: 9^0C (45% concentration)

Boiling point: 136-137^0C (45% concentration)

Vapor pressure: 3.24 mmHg (20^0C, 45% concentration)

D. Photo graphs of laboratory equipments set up

Steam generator

film evaporator

pH meter TDS meter

E. The procedure for Start up the unit (rising film evaporator):

- Switch on the steam supply.
- Heating steam supply pressure: min. 3 bar, Steam mass flow rate: 3 kg/h
- Fill feed vessel with initial solution (max. 25 L).
- Switch on power supply at the master switch (control cabinet).
- Switch on the vacuum pump P_2 and open the gas ballast valve. (Set to required vacuum using vacuum controller).
- Slightly open feed valve and fill rising film evaporator with feed solution using return pipe.
- When the liquid level is visible in the cyclone separator, close the feed valve.
- Set the required heating steam pressure p1 on the controller.
- Switch on the water supply. Using valve set a volumetric flow of ~ 250...300 l/h on the flow meter f2.
- Open valve pressure manometer p_3 increases to the heating steam supply pressure.
- Allow the system to heat up until a mixture of steam and liquid continuously flows from the evaporator into the cyclone. The liquid condenses in the condenser and drips into the condensate tank.
- Slowly open the feed valve and set a constant feed flow rate on the flow meter f1.
- Slowly open valve and set a constant outflow of concentrate into the concentrate tank until the liquid level in the cyclone separator remains constant.
- The user must set the heating steam pressure, the cooling water pressure and the evaporator pressure. Time measurement for the experiment begins from the time at which the first condensate is formed in the condenser.
- From the start of time measurement, collect the outflow of heating steam condensate in a suitable tank and set a constant concentrate outflow and a constant feed inflow.
- Regularly record all temperatures and pressures throughout the experiment.
- Shut off the steam supply shortly before the selected time period has elapsed.

- Stop time recording when no more condensate is formed in the condenser. Stop heating steam condensate quantity measurement, concentrate removal and feed inflow.