Study, Investigation and Control of Caustic Soda Production from Red Sea Water

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B.Sc. (Honours) in Chemical Engineering Technology
University of Gezira, (2014)

A Dissertation
Submitted to the University of Gezira in Partial Fulfillment of the Requirements for the Award of the Degree of Master of Science

In
Chemical Engineering
Department of Chemical Engineering and Chemical Technology
Faculty of Engineering and Technology

March 2017
Study, Investigation and Control of Caustic Soda Production from Red Sea Water

Emadeldeen Abuobaida Mohamed Hamouda

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Date: March/2017
Study, Investigation and Control of Caustic Soda Production from Red Sea Water

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Date of Examination: 21/3/2017
DEDICATION

To my family for their unconditional love.
To my friends for their support.
ACKNOWLEDGMENTS

Above all, I render my thanks to the merciful “Allah” who offered me health and patience to accomplish this study. This research work would have never been successfully undertaken without the unreserved support of my main supervisor Prof. Gurashi Abdella Gasmelseed. I would like to express my deepest gratitude to Eng. Alsunni Osman Hassan Alsunni for his rigorous interest and for sharing me her profound knowledge and experience. His continued discussion and critical comments helped me a lot to improve and refine the final draft of the thesis. Sincere thanks are extended to my family for their help and cooperation in various stages of this work. Last but not least, thanks go to everyone, who contributed to this work.
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Abstract
Study, Investigation and Control of Caustic Soda Production from Red Sea Water

Emadeldeen Abuobida Mohamed Hamouda

Abstract

Control systems are used to maintain process conditions at their desired values by manipulating certain process variables to adjust the variables of interest. The process of electrolysis of red sea water including mixer, electrolysis cell and Multi-Effect Evaporators. A mixer is used to increase the concentration of the sodium chloride in sea water by addition process, the concentrated solution electrolyzed in an electrolysis cell producing liquid caustic soda, hydrogen and chlorine are byproducts. Multi-Effect Evaporators are used to increase the concentration of liquid caustic soda output. The aims of this study are to investigate the production of liquid caustic soda by electrolysis process, automatic control of the Mixer and Multi-Effect Evaporators and analyze the stability of the Mixer and Multi Effect Evaporators using different methods. A single input/single output (SISO) system was used to control the composition of the solution inside the Mixer, composition of caustic soda and the pressure in the fourth evaporator. A feedback control system was used to control the system. The characteristic equation, the open loop transfer function (OLTF) and the overall transfer function were obtained for stability analysis and tuning. The methods used for tuning and stability analysis were Routh-Hurwitz, direct substitution, Root-Locus and Bode plot to determine the ultimate gain ($K_u$) and the ultimate period ($P_u$). The response of the system was plotted by inserting the adjustable obtained parameters. MATLAB documentation (m file) was used to determine the system stability analysis and tuning. The results of this study were for three loops, for composition of the solution inside the mixer control loop1 the average ultimate gain $K_u$ is 11.2 and the average ultimate period is $P_u$ 5.98 sec, for the fourth evaporator at multi-effect evaporators pressure control loop2 the average ultimate gain $K_u$ is 14.4 and the average ultimate period $P_u$ is 9.48 sec, for composition in the fourth evaporator at multi-effect evaporators control loop3 the average ultimate gain $K_u$ is 20.54 and the average ultimate period $P_u$ is 4.52 sec. These methods are proved to be identical in the results and each of them is qualified to be used for tuning and stability analysis without loss of accuracy. It is recommended that the feedback control system should be used to control the composition and pressure of the fourth evaporator.
ملخص الدراسة
دراسة.استقصاء و التحكم لإنتاج الصودا الكاوية من مياه البحر الاحمر
عماد الدين ابو عبدة محمد حمودة

ملخص الدراسة

تستخدم أنظمة التحكم المحاكاة على طريقة العملية عند القيام المطلوبة، من خلال معالجة متغيرات العملية لضبط هذه المتغيرات عند القيام المطلوبة. عمليات التحليل الكهربائي لحياة البحر الأحمر تشمل خليط تحليل كهربائي وميثاق معدات متعددة الأثار. الخلاط يتم زيادة تركيز كلوورد الصوديوم في مياه البحر عن طريق الإضافة. المحلل المركزي يتم تحليله في خليط التحليل الكهربائي لنتاج الصودا الكاوية السائقة. غاز الهيدروجين والكلور منتجات ثانوية لعملية التحليل. المبخرات متعددة الأثار استخدمت لزيادة تركيز محلول هيدروكسيد الصوديوم الناتج. تهدف هذه الدراسة إلى بحث دراسة إنتاج مادة الصودا الكاوية السائقة عن طريق عملية التحليل الكهربائي و التحكم الآلي في الخلاط و المبخرات متعددة الأثار. وتحلية الاستقرارية للخلاط المبخرات متعددة الأثار. تم استخدام النظام ذو الاندلاع والانزلاق الإحالي و ذلك للتحكم في تركيب محلول داخل الخلاط و تركيب نواتج المبخرات متعددة الأثار و الضغط في المبخر الربع. تم استخدام نظام التغذية العكسية ذو الانزلاق و الانزلاق الإحالي للتحكم في النظام. تم وضع استراتيجية للتحكم. وتم إعداد التوابع المنافقة كما جدنت المعادلات المميزة و التوابع المنافقة الكليه و معادلات الدورات المفتوحة حيث استخدمت في الضبط و تحديد الاستقرارية و المحاكة للنظام. استخدمت أربعة طرق للضبط وتحلية الاستقرارية هي روث-هورويتز و التعويض المباشر و جذور لوكس و مخطط بودي لتحديد المكاسب الحرجة و الفترات الحرجة. وقد تم رقم استجابة النظام باستخدام المتغيرات القابلة للتحديث. أيضا استخدم برنامج متالاب لتحديد استقرار النظام وضبطه. و كانت النتائج لهذه الدراسة لمثلثة دونون للتحكم، الدائرة الإحالي للتحكم في تركيب محلول داخل الخلاط و وجد ان متوسط المكاسب الحرجة 11.2 و متوسط الفترات الحرجة 5.9 ثانية، الدائرة الثانية للتحكم في ضغط المفاعل الربع و وجد ان متوسط المكاسب الحرجة 14.4 و متوسط الفترات الحرجة 48.9 ثانية، الدائرة الثالثة للتحكم في تركيب المبخر الربع في المبخرات متعددة الأثار. و وجد ان متوسط المكاسب الحرجة 20.5 و متوسط الفترات الحرجة 45.2 ثانية. اثبتت هذه الطرق تطبيق كبير في النتائج. و وكل منها يستخدم لضبط وتحلية استقرارية النظام دون فقدان الدقة. لذلك أوصى الدراسة بأن يتم استخدام نظام التغذية العكسية ذات الاندلاع و الانزلاق الإحالي للتحكم في ضغط و تركيب المبخر الربع.
CHAPTER ONE

Introduction
Chapter One
Introduction

1.1 Caustic Soda:

Caustic soda is the usual commercial name for sodium hydroxide NaOH and its solution. It gains the importance from its wide use; caustic soda and chlorine are one of the most important heavy chemical industries. It’s obvious to recognize the essential and vital role of the caustic soda by tracing its annual production which it’s about 700000 tonnes for United Kingdom, 11 million tonnes for Europe and 57 million all over the world, that’s shows the importance of NaOH industry. These chemicals rank close to sulfuric acid and ammonia magnitude of dollar value of use. The applications are so diverse that hardly a consumer product is sold that is not dependent at some stage in its manufacture on chlorine and alkali’s [1].

Sodium hydroxide derives from sodium carbonate, formerly named “caustic soda”. In Ancient Egypt, sodium carbonate was already mixed with lime to synthetize an alkali: the hydroxide ion OH\(^{-}\) in solution with the sodium ion Na\(^{+}\). Through the ages, several processes were developed to synthetize it, such as the Solvay process in 1861. Today, sodium hydroxide is mostly produced by the electrolysis of a solution of sodium chloride [1].

The most common process for the manufacture of caustic soda is the electrolysis of sodium chloride brine. The electrolytic processes produce a caustic soda solution that has to be concentrated by evaporation, hydrogen and chlorine are byproducts. This evaporation process is difficult since caustic soda solutions have a high boiling point elevation (BPE). At 50% concentration the BPE is about 80°F (45°C). This limits the number of effects usually to four, with the evaporator operated in reverse flow so that the highest concentration is on the first effect. This effect will typically operate at over 260°F (125°C).

An additional problem is that at high temperature, caustic soda solutions corrode stainless steel. The first and second effect calandrias are usually fabricated in nickel, which is resistant to corrosion. The third and the fourth effects can be Avesta 254SLX, which is far less expensive than nickel. The vapor side of the evaporator can be 304 stainless steel and sometimes carbon steel. Tubular falling film evaporators have been the standard for this application. In recent years, The Adjusted Present Value (APV) has employed a plate evaporator for caustic soda. The plate employs nickel welded pairs and proprietary gaskets. The APV design for caustic soda has proven to be the best solution
to minimize nickel pickup, which is important to the bleach manufacturing industry [1].

1.2 Manufacture of Caustic Soda:-

Caustic soda manufactured or produced by two methods:

1. Produce caustic soda from soda ash and lime.

2. Produce caustic soda by electrolysis of sodium chloride (brine solution) and electrolysis of molten sodium chloride.

1.3 Uses of Caustic Soda:-

Almost all caustic soda is major chemical raw material finding extensive use in many segments of industry such as:

1. In textile processing caustic soda is used for mercerization of cotton, when caustic soda is applied to cotton and then washed out while the cloth is under tension the crystalline orientation of the fiber is changed, this results in fabric having improved luster.

2. In petroleum refining caustic soda is used to remove sulfur compounds such as H₂S and mercaptans.

3. In food processing caustic soda is used to peel fruits and vegetables, to process olives and to refine vegetable oils.

4. In alumina production caustic soda is used to dissolve bauxite as first step in the production of aluminum.

5. In glass production sodium hydroxide can be used to replace part of the soda ash as source of Na₂O the foregoing examples are not intended to be complete and do not cover very large uses of NaOH in the production of specific chemicals.

6. The traditional uses in the fields of soap, manufacture of pulp, production of detergents, rayon, dyes, drugs, duds, rubber textiles, chemicals, bleaching, petroleum and explosives.

7. We use the caustic soda in pulp and paper production [1].
1.4 Control System:-

The technological explosion of the twentieth century, which was accelerated by the advent of computers and control systems, has resulted in tremendous advances in the field of science.

Control systems are an interdisciplinary field covering many areas of engineering and sciences. They exist in many systems of engineering, sciences and human body. Control means to regulate, direct, command, or govern. A system is a collection, set, or arrangement of elements (sub systems) [3].

A Control system is an interconnection of components forming a system configuration that will provide a desired system response. Hence, a control system is an arrangement of physical components connected or related in such a manner as to command, regulate, direct or govern itself or another system [4].

In order to identify, delineate, or define a control system, we introduce two terms: input and output. Here, the input is the stimulus, excitation, or command applied to a control system, and the output is the actual response resulting from a control system. The output may or may not be equal to the specified response implied by the input. Inputs could be physical variables or abstract ones such as reference, set point or desired values for the output of the control system. Control system can have more than one input or output. The input and output represent the desired response and the actual response respectively. A control system provides an output or response for a given input or stimulus, as shown in fig (1.1).

![Fig. 1.1: Description of a control system](image)

If the output and input are known, it is possible to identify or define the nature of the system’s components. In general there are three basic types of control systems:

1. Man-made control systems.
2. Natural including biological-control systems.
3. Control systems whose components are both man-made and natural.

The design of control systems is the selection and arrangement of control system component to perform prescribed task and the analysis of control systems is investigation of the properties and performance of existing control systems [4]. Control systems engineering consists of analysis and design of control systems configurations. Control systems are dynamic, in that they respond to an input by first undergoing a transient response before attaining a steady-state response which corresponds to the input.

There are three main objectives of control systems analysis and design. They are:

1. Producing the response to a transient disturbance which is acceptable.
2. Minimizing the steady-state errors.
3. Achieving stability.

Control systems must be designed to be stable. Their natural response should decay to a zero values as time approaches infinity, or oscillate. The design and analysis of dynamic control systems are based on accurate mathematical models of complex physical systems. These mathematical models, which follow from the physical laws of the process, are generally highly coupled nonlinear differential equations. Definition of stability an unconstrained linear system is said to be stable if the output response is bounded for all bounded input, otherwise it is said to be unstable. By a bounded input, we mean an input variable that stay within upper and lower limit for all values of time.

Automatic control systems enable a process to be operated in safe and profitable method. Control systems are focusing on safety that is including the safety of people, the environment and equipment [4].

1.5 Type of Controllers:

Process control is the measurement of a process variable, the comparison of that variables with its respective set point, and the manipulation of the process in a way that will hold the variable at its set point when the set point changes or when a disturbance changes the process. One way to improve the step response of a control system is to add a controller to the feedback control system, the closed loop systems can be controlled by Proportional control, Proportional Integral (PI) control and Proportional plus Integral plus Derivative (PID) control [8].

1.5.1 Proportional Control:
The proportional action is responds quickly to changes in error deviation; however the proportion controller does not guarantee a zero steady state control error. The proportional controller is consider to be simple controller which is the best and can be used when non zero steady state error is acceptable or if the controlled system contain pure integrator. Generally, it used in pressure control or level control [8].

1.5.2 Proportional Integral (PI) Control:-

The reset (or integral) contribution from more mathematical point of view, at any time the rate of change of the output is the gain time the reset rate times the error is acceptable or if the controlled system contains pure integrator . Generally, it used in pressure control or level control [8].

1.5.3 Proportional Plus Integral Plus Derivative (PID) Control:-

The PID control algorithm is made of three basic responses, proportional (or gain), integral (or reset), and derivative. Derivative is the third and final element of PID control. Derivative responds to the rate of change of the process (or error). Derivative is the normally applied to the process only. Analog PID controllers are common in many applications. They can be easily constructed using analog devices such as operational amplifiers, capacitors and resistors. They are reliable in mechanical feedback systems and able to satisfy many control problems [8].

1.6 Control Classification:-

Control is first classified as being either manual or automatic. This division generally refers to the amount of human effort needed to achieve a common function [8].

1.6.1 Manual Control:-

Manual control is voluntarily initiated within the system with very little human effort. The terms open-loop and forward-feed are frequently used to describe manual control systems. Valve adjustments and switching functions are examples of manual control operations. In general, this type of control is achieved by some degree of physical effort on the part of a human operator [8].

1.6.2 Automatic Control:-
Automatic control by comparison, applies to those things that are achieved, during normal operation, without human intervention. This type of control is used where continuous attention to system operation would be demanded for a long period without interruptions. Automatic control does not, however, necessarily duplicate the type of control achieved by a human operator. Equipment that employs automatic control is limited to only those things that can be forecast by the input data. Terms such as closed-loop control and feedback are commonly used to describe automatic control functions [16].

1.6.3 Open-Loop Control:-

Open-loop control is relatively easy to achieve because it does not employ any automatic equipment to compare the actual output with the desired output. In manufacturing, open-loop operations are achieved by adjustment of the system to some predetermined setting by a human operator. The system then responds to this setting without any modification. Any changes made in operation are based entirely on some outside human judgment to correct the desired output [8].

1.6.4 Closed-Loop Control:-

Closed-loop refers to a type of system that is self-regulating. In this type of system, the actual output is measured and compared with a predetermined output setting. A feedback signal generated by the output sensing component is used to regulate the control element so that the output conforms to the desired value. The term feedback refers to the direction in which the measured output signal is returned to the control element. In a sense, the output of this type of system serves as the input signal source for the feedback control element. Closed-loop control is so named because of the return path created by the feedback loop from the output to the input [8].

1.7 Statement of the Research Problem:-

Generally, the essential problem of the control system in the chemical plants is the control mechanism that will make the proper changes on the process to cancel the negative impact that effect on the desired operation of chemical plant. However, the good performance of the plant is relevant to control mechanism which was used. The aiming is tuning the control loops of the mixer and multi effect evaporators and checking the stability using different methods, in order to develop an appropriate control strategy for the mixer and multi effect evaporators.
1.8 Layout of the Thesis:-

The thesis starts with an abstract that summarizes the methodology and results of the thesis. The thesis is divided to five chapters. Chapter one is an introduction where the general features, research incentives, problem statement and objectives are covered. Chapter two covers the literature Review. Chapter three materials and methods describe the materials that have been used in this work and the overall methodology followed for process control. Chapter four includes the results of the study represented in illustrative graphs and tables. Chapters five summarize the most important results, conclusion and recommendations. All the reference cites in the thesis are presented in the reference list.

1.9 Research Objectives:-

The objectives of this study are:

1. To study, investigate and control the production of liquid caustic soda by electrolysis process.
2. To suggest automatic control of the Mixer and Multi-Effect Evaporators.
3. To analyze the stability of the Mixer and Multi Effect Evaporators using different methods.
CHAPTER TWO

Literature Review
Chapter Two

Literature Review

The process of caustic soda production process developed rapidly by the time. Solvay process or ammonia-soda process is the major industrial process for the production of sodium hydroxide, the ammonia-soda process was developed into its modern form by Ernest Solvay during the 1860s.

The chloralkali process is an industrial process for the electrolysis of NaCl. It is the technology used to produce chlorine and sodium hydroxide (caustic soda). Industrial scale production began in 1892.

2.1 Production of Caustic Soda:

Caustic soda is produced by two processes:

1. Chemical process (soda ash and lime by Solvay process).
2. Electrolytic process (electrolysis of sodium chloride brine and electrolysis of molten sodium chloride) [2].

2.1.1 Production Caustic Soda from Soda Ash (Solvay process):

Caustic soda was originally made by batch wise cauterization of soda (ash) with lime. Na$_2$CO$_3$ + Ca(OH)$_2$ $\rightarrow$ 2NaOH + CaCO$_3$ $\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldOTS
\[ \text{NH}_4\text{OH} + \text{CO}_2 \rightarrow \text{NH}_4\text{CO}_3 \] ...................................................... (2.6)

\[ \text{NH}_4\text{CO}_3 + \text{NaCl} \rightarrow \text{NaHCO}_3 + \text{NH}_4\text{Cl} \] ........................................... (2.7)

\[ 2 \text{NaHCO}_3 + \text{Heat} \rightarrow \text{Na}_2\text{CO}_3 + \text{CO}_2 + \text{H}_2\text{O} \] ........................................... (2.8)

\[ 2\text{NH}_4\text{Cl} + \text{Ca(OH)}_2 \rightarrow 2\text{NH}_3 + \text{CaCl}_2 + \text{H}_2\text{O} \] ........................................... (2.9)

\[ \text{Na}_2\text{CO}_3 + \text{Ca(OH)}_2 \rightarrow 2\text{NaOH} + \text{CaCO}_3 [1, 23] \] ........................................... (2.10)

The electrolytic production of caustic soda known in the eighteenth century, but it wasn’t used until 1890 that caustic was actually produced in this way for industrial consumption. Until shortly before World War 1, the amount of caustic soda sold as a co-product of chlorine from the electrolytic process was almost negligible compared with that made from soda ash lime causticization. In 1940, however, electrolytic caustic began to exceed lime soda caustic and by 1962 the latter had almost disappeared [2].
2.2.1 Production of Caustic Soda by Electrolysis:

The electrolytic process of brine solution which yields chlorine and caustic soda. It has been in use since the 19th century and is a primary industry in the United States, Western Europe, and Japan. There are three primary electrolytic processes for producing chlorine and caustic soda: the diaphragm cell process, the mercury cell process, and the membrane cell process. The first two have been used for over 100 years; the latter was developed in the past 60. The two oldest methods are used the most throughout the world and have been proven to be the most environmentally unfriendly through their use of asbestos and mercury, respectively. The membrane cell process is a superior method in its energy efficiency and lack of harmful chemicals.

The electrolytic processes produce a caustic soda solution that has to be concentrated by evaporation. This evaporation process is difficult since caustic soda solutions have a high boiling point elevation (BPE). At 50% concentration the BPE is about 80°F (45°C). This limits the number of effects usually to three, with the evaporator operated in reverse flow so that the highest concentration in on the first effect. This effect will typically operate at over 260°F (125°C) [1].

An additional problem is that at high temperature, caustic soda solutions corrode stainless steel. The first and second effect calandrias are usually fabricated in nickel, which is resistant to corrosion. The third effect can be Avesta 254SLX, which is far less expensive than nickel. The vapor side of the evaporator can be 304 stainless steel and sometimes carbon steel. Tubular falling film evaporators have been the standard for this application. In recent years, APV has employed a plate evaporator for caustic soda. The plate employs nickel welded pairs and proprietary gaskets. The APV design for caustic soda has proven to be the best solution to minimize nickel pickup, which is important to the bleach manufacturing industry [1].

Almost all Caustic soda manufactured today is produced by the electrolysis of NaCl brines since chlorine is produced as a co-product the technology of caustic soda is intimately bound with that for Cl₂. Chlorine and caustic are produced almost entirely by the electrolysis of aqueous solutions of alkali metal chlorides, or from fused chlorides. Brine electrolysis produces chlorine at the anode and hydrogen along with alkali hydroxide at the cathode.
If chlorine and the alkali hydroxide are to be the final product, cell design must keep them from mixing [1].

### 2.2.2 Energetic of the Electrolytic process:-

Chlorine and sodium hydroxide are prepared by the following serious of reaction:

**Anode:**

\[
2\text{Cl}^- \rightarrow \text{Cl}_2 + 2\text{e}^- \quad \text{.................................................................(2.11)}
\]

\[E^\circ = 1.36\text{v (at 25 °C)}\]

**Cathode:**

\[
2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2 + 2\text{OH}^- \quad \text{.................................................................(2.12)}
\]

\[E^\circ = 0.84\text{v (at 25 °C)}\]

The overall reaction is:

\[
2\text{Na}^- + 2\text{Cl}^- + 2\text{H}_2\text{O} \rightarrow 2\text{Na}^- + 2\text{OH}^- + \text{Cl}_2 + \text{H}_2 \quad \text{.......................................(2.13)}
\]

\[
\Delta G = 421.7\text{ KJ (at 25 °C)}.
\]

The free energy of the of reaction is positive and is provide in the form of electricity. The minimum voltage required for reaction to take place at working temperature of 95 °C is determined as 2.23v. However, this does not take in to consideration the anodic and cathodic over voltages nor the voltage required to overcome the internal resistance of cell [1].
2.2.3 Types of Cells:-

There are three main types of cell:

1. Diaphragm cells.
2. Membrane cells.

Only a few years ago, it seemed that than mercury cell would soon dominate the field because of the high quality of its product and the reduced evaporation required, but unexpected difficulties arose.

In 1979, 50 percent of world production was by mercury cell and 49 percent by diaphragm cells. In United States 74.3 percent of the plants used diaphragm and 20.3 percent mercury cell.

In Japan where mercury cells must be totally replaced by 1984, membrane cells [1].

2.3 Manufacture of Caustic Soda by Electrolysis Process:-

2.3.1 Preparation of Brine:-

A pump is used to suck the water from the sea as a source of weak brine solution the weak solution is to be purified and soften using a thickener and soda ash.

2.3.2 Concentrating of Brine:-

A mixer is used to increase the sodium chloride concentration by adding purified sodium chloride and mixed it to have a thick brine solution with a concentration of 6 percent and then fed to the electrolysis cell.
2.3.3 Electrolysis of Brine:-

The brine solution is fed to membrane electrolysis cell. This technology uses water-impermeable ion-conducting membrane. The membrane is made of a special resin which permits cations (positive ions) to pass through. The anode chamber of a membrane electrolytic cell is filled with brine, and the cathode chamber with water.

The brine in the anode chamber contains sodium (Na+) and chloride (Cl-) ions, these ions migrate when a current is applied: the positively charged sodium ions pass through the membrane to the cathode chamber, while the negatively charged chloride ions are discharged on the anode surface to form chlorine gas (Cl2).

Water in the cathode chamber partly dissociates into hydrogen (H+) and hydroxide (OH-) ions. The hydrogen ions capture electrons on the cathode surface to form hydrogen gas (H2). The hydroxide ions are attracted to the anode, but blocked by the membrane, and react with the sodium ions from the anode chamber to form caustic soda (sodium hydroxide, NaOH) with concentration of 26 percent.

Caustic soda is further concentrated in an evaporators to a concentrated to about 46 percent for delivery.

2.3.4 Evaporation:-

Four multi-effect evaporators are used to concentrate the caustic soda obtained from the cell; this weak solution is concentrated in multiple effect evaporators to produce 46 percent sodium hydroxide. [1, 23]
2.6 Background of Control System:

The technological explosion of the twentieth century, which was accelerated by the advent of computers and control systems, has resulted in tremendous advances in the field of science. Thus, automatic control systems and computers permeate life in all advanced societies today. These systems and computers have acted and are acting as catalysts in promoting progress and development, propelling society into the twenty-first century [4].

Technological developments have made possible high-speed bullet trains; exotic vehicles capable of exploration of other planets and outer space; the establishment of the Alpha space station; safe, comfortable, and efficient automobiles; sophisticated civilian and military [manual and uninhabited] aircraft; efficient robotic assembly lines; and efficient environmentally friendly pollution controls for factories. The successful operation of all of these systems depends on the proper functioning of the large number
of control systems used in such ventures. The action of steering an automobile to maintain a prescribed direction of movement satisfies the definition of a feedback control system [24].

The prescribed direction is the reference input. The driver’s eyes perform the function of comparing the actual direction of movement with the prescribed direction, the desired output. The eyes transmit a signal to the brain, which interprets this signal and transmits a signal to the arms to turn the steering wheel, adjusting the actual direction of movement to bring it in line with the desired direction. Thus, steering an automobile constitutes a feedback control system. One of the earliest open-loop control systems was Hero’s device for opening the doors of a temple. The command input to the system was lighting a fire upon the altar. The expanding hot air under the fire drove the water from the container into the bucket. As the bucket became heavier, it descended and turned the door spindles by means of ropes, causing the counterweight to rise. The door could be closed by dousing the fire. As the air in the container cooled and the pressure was thereby reduced, the water from the bucket siphoned back into the storage container [4].

Thus, the bucket became lighter and the counterweight, being heavier, moved down, thereby closing the door. This occurs as long as the bucket is higher than the container. The device was probably actuated when the ruler and his entourage started to ascend the temple steps. The system for opening the door was not visible or known to the masses. Thus, it created an air of mystery and demonstrated the power of the Olympian gods.

James Watt’s fly ball governor for controlling speed, developed in 1788, can be considered the first widely used automatic feedback control system.

Maxwell, in 1868, made an analytic study of the stability of the fly ball governor. This was followed by a more detailed solution of the stability of a third-order fly ball governor in 1876 by the Russian engineer Wischnegradsky. Minorsky made one of the earlier deliberate applications of nonlinear elements in closed-loop systems in his study of automatic ship steering about 1922 [22].

Significant date in the history of automatic feedback control systems is 1934, when Hazen’s paper ‘Theory of Servomechanisms’ was published in the Journal of the Franklin Institute, marking the beginning of the very intense interest in this new field. It was in this paper that the word servomechanism originated, from the words servant (or slave) and mechanism. Black’s important paper on feedback amplifiers appeared in the same year. After the Second World War, control theory was studied intensively and applications have proliferated.
Many books and thousands of articles and technical papers have been written, and the application of control systems in the industrial and military fields has been extensive. This rapid growth of feedback control systems was accelerated by the equally rapid development and widespread use of computers [22].

2.7 Feedback Control Systems:

Feedback is the property of a closed loop system, which allows the output to be compared with the input to the system such that the appropriate control action may be formed as some function of the input and output [5].

For more accurate and more adaptive control, a link or feedback must be provided from output to the input of an open-loop control system. So, the controlled signal should be fed back and compared with the reference input, and an actuating signal proportional to the difference of input and output must be sent through the system to correct the error. In general, feedback is said to exist in a system when a closed sequence of cause and effect relations exists between system variables. The reference input N reset the desired idle-speed. The engine idle speed N should agree with the reference value N rand any difference such as the load-torque T is sensed by the speed-transducer and the error detector. The controller will operate on the difference and provide a signal to adjust the throttle angle to correct the error [5].

The basic hardware components of the feedback control loop are the following bellow:

![Figure (2.2) Standard block diagram of the feedback control system](image)

Where:

- Y = controlled variable
- U = manipulated variable
- D = disturbance variable (also referred as load variable)
P = controller output
E = error signal
Ym = measured value of Y
Ysp = set point
Yu = change in y due to U
Yd = change in Y due to D
Gc = controller transfer function
Gv = valve transfer function
Gp = process transfer function
Gd = disturbance transfer function
Gm = transfer function for sensor and transmitter

In general, three types of criteria are used to assess closed loop system performance:
1. Stability criteria.
2. Steady state criteria.
3. Dynamic response criteria [6].

2.8 Feed-Forward Control System:

Feed-forward control is a strategy used to compensate for disturbances in a system before they affect the control variable. A feed forward control system measures a disturbance variable, predicts its effect on the process, and applies corrective action [6].
Chapter Three

Materials and Methods

3.1 Introduction:

This chapter contains brief information about the methods for determining optimum conditions to manufacturing of caustic soda by electrolysis process (graphically and analytically) and types of materials used in this research such as: sensors or transmitters, controllers and brief history about MATLAB software. Also the stability tests, tuning controllers and overall system response.

3.2 Optimum Design and Design Strategy:

An optimum design is based on the best or most favorable conditions. These optimum conditions can ultimately be reduced to a consideration of costs or profits. Thus, an optimum economic design could be based on conditions giving the least cost per unit of time or the maximum profit per unit of production. When one design variable is changed, it is often found that some costs increase and others decrease. Under these conditions, the total cost may go through a minimum at one value of the particular design variable, and this value would be considered as an optimum [19].

Although cost considerations and economic balances are the basis of most optimum designs, there are times when factors other than cost can determine the most favorable conditions. For example, in the operation of a catalytic reactor, an optimum operation temperature may exist for each reactor size because of equilibrium and reaction-rate limitations. This particular temperature could be based on the maximum percentage conversion or on the maximum amount of final product per unit of time. Ultimately cost variables need to be considered, and the development of an optimum operation design is usually merely one step in the determination of an optimum economic design [10].

3.3 Evaporators Control:

The control of most chemical industrial evaporators systems is quite simple. With hygienic evaporators the control is somewhat more complicated due to the need to start up, operate, shut down and then clean at quite frequent intervals. As a result sophisticated control is more likely to be needed on hygienic systems [21].
On almost all evaporation systems there are only two basic objectives:

- To concentrate a liquid to a pre-defined solids content.
- To process a pre-defined feed rate of raw material.

Product concentration has been measured using refractive index, density and viscosity techniques.

Over the last ten years, the use of mass flow meters for density measurement has become the standard. This type of meter provides an accurate measurement (usually out to the 4th decimal place) of both flow and density. The density measurement, which is easily converted to solids content, can then be used to control either product removal rate from the evaporator, steam flow, or feed flow.

There are two techniques used to control evaporators, and the choice is based on the design of the evaporator. In applications where liquid recirculation is required to maintain sufficient wetting in the final stage of the evaporator, the product concentration control is simple and accurate. The procedure is to set the steam flow rate at the design value, remove product based on density in the recirculation loop, and adjust the feed flow to maintain liquid levels in the evaporator. When a higher throughput is required, then the steam rate is increased. This technique provides excellent control of the product concentration with conventional analog controllers. For heat sensitive products, it is best to avoid recirculation whenever possible. In the case of once-through-flow in the final stage, there is no recirculation loop in which to install the transmitter and to delay discharge of product when not on specification. In this case, the method is to set the feed flow rate to the desired value and then change the energy input to produce the product concentration required. The energy input may be the steam rate or the power into the MVR. This technique does not control product quality particularly accurately, since response is slow. However it is satisfactory for most purposes and the user can always apply more sophisticated PLC control when necessary [21].

Almost all evaporators will have to be cleaned at some time. Some chemical evaporators may run for months between cleaning cycle. Also with non-hygienic duties, the only requirement is to clean the heat transfer surface sufficiently to restore design performance. In the case of hygienic evaporators, the concern is not only plant operation, but also contamination from bacteria. Typically, a hygienic evaporator will be cleaned
every day. Dairy evaporators, which are designed and constructed to 3A standards, are subject to one of the highest cleaning standards. The inspector will expect that the equipment be cleaned completely with no residue left on any surfaces. The potential labor costs to start up, shut down and go through a complex cleaning cycle, on a daily basis, are very high. A fully automatic system is therefore required to perform all these operations. These functions are ideally performed by a PLC [21].

3.4 Control of the mixer System:-
Liquid–solid mixing is typically done to suspend coarse free-flowing solids, or to break up lumps of fine agglomerated solids. An example of the former is the mixing granulated sugar into water; an example of the latter is the mixing of flour or powdered milk into water. In the first case, the particles can be lifted into suspension (and separated from one another) by bulk motion of the fluid; in the second, the mixer itself (or the high shear field near it) must destabilize the lumps and cause them to disintegrate.

One example of a solid–liquid mixing process in industry is concrete mixing, where cement, sand, small stones or gravel and water is commingled to a homogeneous self-hardening mass, used in the construction industry.

We use the mixer to concentrate the brine solution which obtained from the seawater, feedback control system is used to control the composition of the brine solution inside the mixer.

3.5 Single Input/Output:-
Single Input, Single Output (SISO) systems involves a single loop control that uses only one measured signal (input). This signal is then compared to a set point of the control variable (output) before being sent to an actuator (i.e. pump or valve) that adjusts accordingly to meet the set point.[23].

3.6 Process:-
The material equipment along with the physical or chemical operations which take place (multi-effect evaporators, reactors…etc.) [13].

3.6.1 Measuring Instruments or Sensor:-
For example, thermocouples (for temperature), bellow or diaphragms (pressure or liquid level) [16].
3.6.2 Transmission Lines:-

Used to carry the measurements signal from the sensors to the controller and control signals from controllers to the final element. These lines can be either pneumatic or electrical [23].

3.6.3 Final Control Element:-

This is the device that receives the control signal from the controller and implements it by physically adjusting the value of the manipulated variables. Usually, a control valve or a variable-speed metering pump [23].

3.6.4 Controller:-

Between the measuring device and final control element comes the controller; it is considered as the brain of the control loop. The controller performs the decision operation in the control system. Its function is to receive the measured output signal \( y_m(t) \) and after comparing it with the set point \( y_{sp} \) to produce the actuating signal \( p(t) \) in such a way as to return the output to the desired value \( y_{sp} \). Therefore, the input to the controller is the error \( E(t) = y_{sp} - y_m(t) \), while its output is \( p(t) \) [6].

3.7 System Stability and Tuning:-

Mathematical models of a system have been obtained in transfer function form, and then these models can be analyzed to predict how the system will respond in the both the time and frequency domains [23].

3.7.1 Stability:-

Systems have several properties such as controllability, observability, stability, and inevitability these characteristics; stability plays the most important role.

Dynamically a stable system is the one for which the output is bounded for all bounded inputs. A system exhibiting an unbounded response to a bounded input is unstable. Bounded output is a function of time that always remains between an upper and lower limit [11].

The most basic practical control problem is the design of a closed-loop system such that its output follows its input as closely as possible, unstable systems cannot guarantee such behavior and therefore are not useful in practice. As soon as stability is guaranteed, then one seeks to satisfy other design requirements, such as speed of response, settling time, bandwidth, and steady-state error. The concept of stability has
been studied in depth, and various criteria for testing the stability of a system have been proposed. Among the most celebrated stability criteria are those of Routh, Hurwitz, Nyquist and Bode [23].

System stable if the output remains bounded for all bounded inputs. Practically, this means that the system will not blow up while in operation.

The system can be considering its response to a finite input signal. This means the analysis of the system dynamics in the actual time-domain. Several methods have been developed to deduce the system stability from its characteristic equation. They are “short-cut” methods for providing information without finding out the actual response of the system. All these methods are based on the criterion that a sufficient condition for stability of a control loop is to have a characteristic equation with only negative real roots and/or complex roots with negative real parts. The methods or technique for assessment of the stability of a system include the following:

1. Direct substitution method.
2. Routh-Hurwitz test.

### 3.7.2 The Algebraic Methods:

The algebraic stability criteria are based on the characteristic equation of the system to be analyzed. They contain algebraic conditions as inequalities between coefficients, which are only valid if all roots of the polynomial lie in the left half S-plane [22].

#### 3.7.2.1 The Routh Stability Criterion:

The Routh stability criterion provides a convenient method of determining control systems stability. It’s also determines the number of characteristic roots within the left half, and the number of roots on the S-plane, and the number characteristic roots in the stable left half, and the number of roots on the imaginary axis. It does not locate the roots.

The method may also be used to establish limiting values for a variable factor beyond which the system would become unstable.

Assume the characteristic equation of interest is an N th-order polynomial.

[22].
Table (3.1) Routh Array:

<table>
<thead>
<tr>
<th>Row</th>
<th>Coefficients</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$a_n$ $a_{n-2}$ $a_{n-4}$ $a_{n-6}$</td>
</tr>
<tr>
<td>2</td>
<td>$a_{n-1}$ $a_{n-3}$ $a_{n-5}$ $a_{n-7}$</td>
</tr>
<tr>
<td>3</td>
<td>$b_1$ $b_2$ $b_3$</td>
</tr>
<tr>
<td>4</td>
<td>$c_1$ $c_2$ $c_3$</td>
</tr>
<tr>
<td>5</td>
<td>$d_1$ $d_2$</td>
</tr>
<tr>
<td>6</td>
<td>$e_1$ $e_2$</td>
</tr>
</tbody>
</table>

\[a_n x^n + a_{n-1} x^{n-1} + \cdots + a_2 x^2 + a_1 x + a_0 = 0\]......2.14

The Routh array is formed as given below:

\[
\begin{array}{c|cccc}
 s^n & a_n & a_{n-2} & a_{n-4} & \cdots \\
 s^{n-1} & a_{n-1} & a_{n-3} & a_{n-5} & \cdots \\
 s^{n-2} & b_1 & b_2 & b_3 & \quad \text{(2.10)} \\
 s^{n-3} & c_1 & c_2 & c_3 & \\
 \vdots & & & & \\
 s^0 & q & \\
\end{array}
\]

Routh array =

Where the $a_1, b_1, \ldots$ are calculated from the equations

\[
b_1 = \frac{a_{n-1} a_{n-2} - a_{n-3} a_{n-3}}{a_{n-1}} \quad \text{(2.10)}
\]

\[
c_1 = \frac{b_1 a_{n-3} - b_2 a_{n-1}}{b_1} \quad \text{(2.17)}
\]

Then the first column of the array of equation is examined. The number of sign changes of this column is equal to the number of roots of the polynomial that are in the (RHP).
Frist column = \[
\begin{bmatrix}
  a_n \\
  a_{n-1} \\
  b_1 \\
  c_1 
\end{bmatrix}
\]

Thus for the system to be stable there can be no sign changes in the first column of the Routh array [22]

**3.7.2.2 Direct Substitution Analysis:**

The closed-loop poles may lie on the imaginary axis at the moment a system becomes unstable. We can substitute \( s = i\omega \) in the closed-loop characteristic equation to find the proportional gain that corresponds to this stability limit (which may be called marginal unstable). The value of this specific proportional gain is called the critical or ultimate gain. The corresponding frequency is called the crossover or ultimate frequency[31]. The ultimate gain and ultimate period that can be used in Z-N continuous cycling relations, and the result on ultimate gain is consistent with Routh array analysis and limited to relatively simple systems. [22]

**3.7.2.3 Root Locus Analysis:**

The idea of a root locus plot is simple if we have a computer. We pick one design parameter, say, the proportional gain, and write a small program to calculate the roots of the characteristic polynomial for each chosen value of \( s \) in 0, 1, 2, 3, ... , 100, ... , etc. The results (the values of the roots) can be tabulated or better yet, plotted on the complex plane. Even though the idea of plotting a root locus sounds so simple, it is one of the most powerful techniques in controller design and analysis when there is no time delay. Root locus is a graphical representation of the roots of the closed-loop characteristic polynomial (i.e., the closed-loop poles) as a chosen parameter is varied. Only the roots are plotted [15].

The values of the parameter are not shown explicitly. The analysis most commonly uses the proportional gain as the parameter. The value of the proportional gain is varied from 0 to infinity, or in practice, just "large enough."

Consider the characteristic equation:

\[
1 + \frac{K(s + z_1)(s + z_2)\cdots(s + z_n)}{(s + p_1)(s + p_2)\cdots(s + p_n)} = 0
\]

................................................................................................................. (3.1)
Equation can be written as

$$1 + \frac{K \text{num}}{\text{den}} = 0$$ \hspace{1cm} \text{..........................(3.2)}

Where \text{num} is the numerator of the polynomial and \text{den} is the denominator polynomial, and \text{K} is the gain (\text{K} > 0). The vector \text{K} contains all the gain values for which the closed loop poles are to be computed.

The root locus is plotted by using the MATLAB command

\text{rlocus (num, den).................................(3.3)}

The gain vector \text{K} is supplied by the user.

The matrix \text{r} and gain vector \text{K} are obtained by the following MATLAB commands:

\text{[r, k] = rlocus (num, den)}
\text{[r, k] = rlocus (num, den, k)}
\text{[r, k] = rlocus (A, B, C, D)}
\text{[r, k] = rlocus (A, B, C, D, K)}
\text{[r, k] = rlocus (sys)}

The following MATLAB commands are used for plotting the root loci with mark ‘0’ or ‘x’:

\text{r = rlocus (num, den).................................(3.4)}

Plot (r, ‘0’) or plot (r, ‘x’).

\text{Figure(3.1) Root Locus}

A root locus plot is a figure that shows how the roots of the closed loop characteristic equation vary as the gain of the feedback controller changes from zero to infinity. The abscissa is the real part of the closed loop root; the ordinate is the imaginary part. Since
we are plotting closed loop roots, the time constants and damping coefficients that we pick off these root locus plots are all closed loop time constants and closed loop damping coefficients [15].

- Root locus is the plots, in complex plane, of the roots of the OLTF.

- They are very useful to determine the stability of closed loop system as the gain changes.

The value of the frequency which take from the Root locus plot used to calculate the limit gain and ultimate period.

One absolute method of determining whether complex or real roots lie in the right hand plane is by use of Routh’s criterion the method entails systematically generating a column of numbers that are then analyzed for sign variations. The first step is to arrange the denominator of the transfer function into descending powers of s. All terms including those that are zero should be included.[11]

3.7.2.4 Bode Plots:-

Bode plots require two curves to be plotted instead of one. This increase in the number of plots is well worth the trouble because complex transfer functions can be handled much more easily using Bode plots. The two curves show how magnitude ratio and phase angle (argument) vary with frequency.

Bode diagrams are rectangular plots. Bode diagram are also known as logarithmic plot and consist of two graphs: the first one is a plot of the logarithmic of the magnitude of a sinusoidal transfer function; the second one is a plot of the phase angle. Both these graphs are plotted against the frequency on a logarithmic scale. The MATLAB command “bode” obtains the magnitudes and phase angles of the frequency response of continuous time, linear, time invariant systems.

The MATLAB Bode commands commonly used are:
Bode (num, den)
Bode (num, den, w)
Bode (A, B, C, D)
The value of the frequency which take from the Bode plot used to calculate the limit gain and ultimate period [11].

3.8 Tuning Controllers:-

Tuning means setting the adjustable parameters (proportional band/gain, integral gain/reset, derivative gain/rate) of a controller to give best performance. For the PID controller [23].

3.8.1 Ziegler – Nichols Tuning Technique:-

The Ziegler-Nichols (ZN) controller settings (.I. G. Ziegler and N. B. Nichols, Trans. ASME 64: 759, 1942) are pseudo-standards in the control field. They are easy to find and to use and give reasonable performance on some loops. The ZN method consists of first finding the ultimate gain Kc the value of gain at which the loop is at the limit of stability with a proportional-only feedback controller. The period of the resulting oscillation is called the ultimate period, Pu (minutes per cycle). The ZN settings are then calculated from Ku and Pu by the formulas [23].

Given in Table for the three types of controllers. Notice that a lower gain is used when integration is included in the controller (PI) and that the addition of derivative permits a
higher gain and faster reset.

The Z-N method is based on frequency response analysis. Unlike the process reaction curve method which uses data from the open-loop response of system. The Z-N tuning technique is a closed-loop procedure [18].

- Bring the system to the desired operation level (design conditions).
- Using P-control only, and with the feedback-loop closed, introduce a sinusoidal set point changing with low amplitude and varying frequency until the system oscillates continuously, the frequency of continuous oscillation is the crossover frequency, $\omega_c$. Let $M = \text{amplitude ratio of the system's response at the crossover frequency.}$
- Ultimate gain $= k_u = 1/M$
- Ultimate period

Table (3.2): Ziegler-Nichols adjustable controller parameters for additives loop for Routh-Hurwitz,[23]

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>$K_c$</th>
<th>$\tau_i(\min)$</th>
<th>$\tau_d(\min)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>0.5$K_u$</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>0.45$K_u$</td>
<td>$\frac{pu}{1.2}$</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>0.6$K_u$</td>
<td>$\frac{pu}{2}$</td>
<td>$\frac{pu}{8}$</td>
</tr>
</tbody>
</table>

3.9 Time Response:-

The time response represents how the state of dynamic system changes in time when subjected to particular input. Since the models have been derived consist of differential equations, some integration must be performed in order to determine the time response of the system. Fortunately, MATLAB provides many useful resources for calculating time responses for many types of inputs.

MATLAB provides tools for automatically choosing optimal PID gains. The tuning algorithm directly using “pidtune” or through using “pidtool”[23].

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3.10 Sensors and Measuring Devices:

The successful operation of any feedback control system depends in a very critical manner. There are different commercial measuring devices which differ either in the basic measuring principle or their construction characteristics [8].

Table (3.3) lists typical measuring devices encountered in various applications of process control [8]:

<table>
<thead>
<tr>
<th>Comments</th>
<th>Measuring device</th>
<th>Measuring process variable</th>
</tr>
</thead>
<tbody>
<tr>
<td>Most comment for relatively low temperature</td>
<td>Thermocouple Resistance thermometer Radiation pyrometer</td>
<td>Temperature</td>
</tr>
<tr>
<td>Used for high temperature</td>
<td>Oscillating quartz crystal</td>
<td></td>
</tr>
<tr>
<td>With floats or displaces</td>
<td>Manometers Bourdon – tube elements Diaphragm elements</td>
<td>Pressure</td>
</tr>
<tr>
<td>Used to convert pressure to electrical signal</td>
<td>Strain pages Piezoresistivity elements Piezoelectric elements</td>
<td></td>
</tr>
<tr>
<td>Measuring pressure drop across flow constriction. For high precision</td>
<td>Orifice plate Venture flow Nozzle flow Turbine flow meters Ultrasound Hot-wire anemometry</td>
<td>Flow</td>
</tr>
<tr>
<td>Couple with various types of indicators and signal converters Good for system with two phases</td>
<td>Float-actuated devices Displacer devices</td>
<td>Liquid level</td>
</tr>
<tr>
<td></td>
<td>Infrared analyzers</td>
<td>Ultraviolet analyzers</td>
</tr>
<tr>
<td>-------------------------</td>
<td>-------------------------------</td>
<td>-------------------------------</td>
</tr>
<tr>
<td>Long times required for analysis.</td>
<td>Conventional for one or two chemicals.</td>
<td>Not very convenient for process control</td>
</tr>
</tbody>
</table>

### 3.11 MATLAB Software:

The name MATLAB is an acronym for matrix laboratory. MATLAB software has become popular in engineering fields as result it has been considered as the world standard for performing simulation and analyzing linear and nonlinear dynamic systems. MATLAB was developed primarily by Cleve Moler in 1970’s. It combines nicely calculations and graphic plotting. MATLAB also integrates programming, visualization and computation in an easy to use environment. MATLAB is a high level technical computing environment suitable for solving scientific and engineering problems. MATLAB features a family of applications-specific solutions called toolboxes. Toolboxes are comprehensive collection of MATLAB functions (M-file) that extend the MATLAB environment to solve particular classes of problems. Areas in which toolboxes are available including signal processing, control systems, neural networks, fuzzy logic wavelets, simulation and many others. The control system toolboxes are a collection of algorithms, written mostly as M-file, that implement common control system design, analysis and modeling techniques [12].
CHAPTER FOUR

Results and Discussion
Chapter Four

Results and Discussions

4.1 Control Strategy:-

A single input/single output (SISO) system was used to control the composition of sodium chloride at the mixer.

A single input/single output (SISO) system was used to control the composition of caustic soda at the fourth evaporator.

A single input/single output (SISO) system was used to control of the pressure at the fourth evaporator.

4.1.1 Sedimentation Process:-

![Figure (4.1) Sedimentation process]

4.1.2 Increase the concentration of the sodium chloride:-

![Figure (4.2) Mixer]
4.1.3 Electrolysis process:

Figure (4.3) an electrolysis cell

4.1.4 Evaporation process:

Figure (4.4) Multi-Effect Evaporators
4.2 Control of Mixer:

![Physical diagram for composition control loop 1](image)

**Figure (4.5) Physical diagram for composition control loop 1**

4.2.1 Composition Control Loop Transfer Function:

![General block diagram of the conventional loop1 feedback control](image)

**Figure (4.6) General block diagram of the conventional loop1 feedback control**

4.2.2 Over all Transfer Function of Loop (1):

\[ G_C = K_C \] \hspace{1cm} (4.1)

\[ G_V = \frac{1}{1.3S+1} \] \hspace{1cm} (4.2)

\[ G_P = \frac{0.4}{1.5S+1} \] \hspace{1cm} (4.3)
\[ G_M = \frac{1}{S+1} \] \hfill (4.4)

\[ 1 + \frac{0.4K_C}{(1.5S + 1)(1.3S + 1)} = 0 \] \hfill (4.5)

\[ (s+1)(1.5s+1)(1.3s+1) + 0.4K = 0 \] \hfill (4.6)

\[ 1.95 s^3 + 4.75 s^2 + 3.8 s + 1 + 0.4K_C = 0 \] \hfill (4.7)

The chr.eq:

\[ \frac{1}{S+1} + \frac{Y_{sp}}{Y(S)} \frac{4}{1.5S + 1} + \frac{Y}{Y(S)} \] \hfill (4.8)

Using Routh array:

Number of rows=4

\[
\begin{bmatrix}
S^3 & 1.95 & 3.8 \\
S^2 & 4.75 & 1+0.4K_C \\
S^1 & b_1 & b_2 \\
S^0 & c_1 & c_2
\end{bmatrix}
\]
This system is stable.

### 4.2.4 Using Direct Substitution Method:

Obtain the frequency:

The Characteristic equation:

\[ 1.95 s^3 + 4.75 s^2 + 3.8 s + 1 + 0.4K_C = 0 \] \hspace{1cm} (4.8)

Set \( S = i\omega \), \( (i^2 = -1) \):

\[-1.95 i\omega^3 - 4.75\omega^2 + 3.8 i\omega + 1 + 0.4 K_C = 0 \] \hspace{1cm} (4.12)

Taking imaginary part of this loop = 0

\[-1.95 i\omega^3 + 3.8 i\omega = \] \hspace{1cm} (4.13)

\[ \omega^2 = 1.94 \]

\[ \omega = 1.39 \text{ rad/s} \]

Taking the real part of loop = 0

\[-4.75\omega^2 + 1 + 0.4K_C = 0 \] \hspace{1cm} (4.14)
\[-4.75(1.39)^2 + 1 + 0.4K_C = 0\]

\[K_C = K_u = 20.44\]

\[P_u = 2\pi / \omega_{co}\]

\[P_u = 2\pi / 1.39 = 4.52 \text{sec}\]

4.2.5 Apply the Root–Locus Using MATLAB:

Plot Root–Locus:

\[\text{OLTF} = \frac{0.4K_C}{(1.5s+1)(s+1)(1.38+1)}\] \hspace{1cm} \text{...(4.15)}

MATLAB Form:

\[\text{>> num=[0.4];}\]

\[\text{>> a=conv([1.3 1],[1 1]);}\]

\[\text{>> den=conv(a,[1.5 1]);}\]

\[\text{>> sys=tf(num,den)}\]

Transfer Function:

\[\frac{0.4}{1.955^2 + 4.755^2 + 3.85 + 1}\]

\[\text{>> rlocus(sys)}\]
$K_u = 20.3$

$\omega = 1.39$ rad/sec

$P_u = \frac{2\pi}{0.662} = 4.52$ sec

### 4.2.6 System Stability Using Bode Method:

Determination of ultimate period ($P_u$) and ultimate gain ($K_u$).

$$OLTF = \frac{0.4Ke}{(1.5s+1)(s+1)(1.3s+1)} \quad (4.16)$$

MATLAB commands:

```matlab
>> a=conv([1.3 1],[1 1]);
>> den=conv(a,[1.5 1]);
>> sys=tf(num,den)
```
Transfer function:

\[
\frac{0.4}{(1.95s^3 + 4.75s^2 + 3.8s + 1)}
\]

>> bode(sys),grid

Figure (4.9) Bode diagram for loop (1)

\(\omega = 1.39 \ \text{rad/sec}\)

\[\text{magnitude(dB)} = -26.2\]

\[20\log AR = dB\]

\[20\log AR = -26.2\]

\(AR = 0.048\)

\(K_u = 1/AR\)

\(K_u = 1/0.048 = 20.83\)

\(P_u = 2\pi/1.39 = 4.52\text{sec}\)
4.2.7 Determination of adjustable parameters:

The average $\langle P_{u,av} \rangle = (P_{u,1} + P_{u,2} + P_{u,3}) / 3 = 4.52$ sec

The average $\langle K_{u,av} \rangle = (K_{u,1} + K_{u,2} + K_{u,3} + K_{u,4}) / 4 = 20.54$

Table (4.1): Ziegler-Nichols adjustable controller parameters for additives loop for Routh-Hurwitz[23]

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>$K_c$</th>
<th>$\tau_i$ (min)</th>
<th>$\tau_d$ (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>0.5Ku</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>0.45Ku</td>
<td>$\frac{pu}{1.2}$</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>0.6Ku</td>
<td>$\frac{pu}{2}$</td>
<td>$\frac{pu}{8}$</td>
</tr>
</tbody>
</table>

Table (4.2) Z-N tuning parameters of loop 1

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>$K_c$</th>
<th>$\tau_i$ (min)</th>
<th>$\tau_d$ (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>10.27</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>9.243</td>
<td>3.76</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>8.64</td>
<td>2.26</td>
<td>0.565</td>
</tr>
</tbody>
</table>

4.2.8 Response of the System for P Controller:

$Y_S = \pi_P / (1 + \pi_L)$

$\pi_P = \frac{0.4K_c}{(1.5s+1)(1.3s+1)}$..............................(4.89)

$1 + \pi_L = 1 + \frac{0.4K_c}{(1.5s+1)(s+1)(1.3s+1)}$..............................(4.54)

$Y_S = \frac{0.4K_c(s+1)}{-19.5 s^2 - 47.5 s^2 + 3.8 s + 1 + 0.4K_c}$..............................(4.5)
Putting $K_C = 10.27$

\[ Y_3 = \frac{(4.10^5 + 4.10)}{1.95s^2 + 4.75s^2 + 3.8s + 5.10} \] \hspace{1cm} (4.85)

MATLAB former:

\[
\text{>> num=[4.10 4.10];} \\
\text{>> den=[1.95 4.75 3.8 5.10];} \\
\text{>> step(num,den),grid}
\]

Figure (4.10) Step Response Of loop 1 for P Controller.

Table (4.3) Simulation Parameter of Loop 1 for P Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>1.15</td>
</tr>
<tr>
<td>Beak time</td>
<td>2.64</td>
</tr>
<tr>
<td>Settling time</td>
<td>38.4</td>
</tr>
<tr>
<td>Over shoot</td>
<td>1.56</td>
</tr>
</tbody>
</table>
Decay Ratio $\frac{1.18}{1.56} = 0.75$

4.2.9 System Response for PI-Controller:

From (Z-N) table for PI-Controller

$$K_c = 9.243 \quad \tau_i = 3.76$$

$$\text{num} = [34.75 \ 3.76];$$
$$\text{den} = [3.76 \ 0];$$
$$m = \text{tf}(\text{num}, \text{den});$$
$$\text{num1} = 1;$$
$$\text{den1} = [1.3 \ 1];$$
$$r = \text{tf}(\text{num1}, \text{den1});$$
$$\text{num2} = 0.4;$$
$$\text{den2} = [1.5 \ 1];$$
$$w = \text{tf}(\text{num2}, \text{den2});$$
$$\text{num3} = 1;$$
$$\text{den3} = [1 \ 1]$$
$$x = \text{tf}(\text{num3}, \text{den3});$$
$$z = \text{feedback}(m \cdot r \cdot w, x)$$
$$\text{step}(z)$$

Transfer function

$$\frac{13.95s^2 + 15.45 + 1.504}{7.382s^4 + 17.86s^3 + 14.29s^2 + 17.66s + 1.504}$$
Table (4.4) Simulation Parameter of Loop 1 for PI Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>0.986</td>
</tr>
<tr>
<td>Beak time</td>
<td>2.69</td>
</tr>
<tr>
<td>Settling time</td>
<td>30.1</td>
</tr>
<tr>
<td>Overshoot</td>
<td>1.59</td>
</tr>
</tbody>
</table>

Decay Ratio $= \frac{1.21}{1.59} = 0.761$
4.2.10 System Response for PID-Controller:

From (Z-N) table for PI-Controller

\[ K_c = 8.64 \quad \tau_i = 2.26 \quad \tau_d = 0.565 \]

\[
\text{num} = [11.03 \ 19.52 \ 8.64]; \\
\text{den} = [0.565 \ 0]; \\
\text{m} = \text{tf}(\text{num}, \text{den}); \\
\text{num1} = 1; \\
\text{den1} = [1.3 \ 1]; \\
\text{r} = \text{tf}(\text{num1}, \text{den1}); \\
\text{num2} = 0.4; \\
\text{den2} = [1.5 \ 1]; \\
\text{w} = \text{tf}(\text{num2}, \text{den2}); \\
\text{num3} = 1; \\
\text{den3} = [1 \ 1]; \\
\text{x} = \text{tf}(\text{num3}, \text{den3}); \\
\text{z} = \text{feedback}(\text{m} \times \text{r} \times \text{w}, \text{x}); \\
\text{step}(\text{z})
\]

Transfer function = \[
\frac{-4.4125s^8 + 12.22s^6 + 11.26s^4 + 3.456}{1.102s^8 + 2.684s^6 + 6.559s^4 + 8.373s^2 + 3.456}
\]
Figure (4.12) Step Response Of loop 1 for PID Controller

Table (4.5) Simulation Parameter of Loop 1 for PID Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>0.201</td>
</tr>
<tr>
<td>Peak time</td>
<td>0.943</td>
</tr>
<tr>
<td>Settling time</td>
<td>12.4</td>
</tr>
<tr>
<td>Over shoot</td>
<td>2.51</td>
</tr>
</tbody>
</table>

Decay Ratio = \(\frac{2.52}{1.52} = 0.60\)
4.2.11 Response of the System for P, PI and PID Controller:

num = [4.10 4.10];
den = [1.95 4.75 3.8 5.10];
sys = tf(num, den);
step(num, den)
hold
num1 = [13.9 15.4 1.504];
den1 = [7.332 17.86 14.29 17.66 1.504];
sys1 = tf(num1, den1);
step(num1, den1)

num2 = [4.412 12.22 11.26 3.456];
den2 = [1.102 2.684 6.559 8.373 3.456];
sys2 = tf(num2, den2);
step(num2, den2)

Blue = P, Green = PI, Red = PID

Figure (4.13) Step Response Of loop 1 for P, PI and PID Controller.
4.3 Control of pressure in the Multi-effect evaporator:

Figure (4.14) Physical diagram for pressure control loop 2

4.3.1 Pressure Control Loop 2 Transfer Function:

Figure (4.15) General block diagram of the conventional loop2 feedback control
4.3.2 Overall Transfer Function of Loop (2):

\[ G_C = K_C \] .......................................................... (4.1)

\[ G_V = \frac{1}{s+1} \] ..................................................... (4.2)

\[ G_P = \frac{1}{5s+1} \] ....................................................... (4.3)

\[ G_M = \frac{1}{5s+1} \] ....................................................... (4.4)

![Figure 4.16](image)

**Figure (4.16) Conventional loop 2 feedback control**

4.3.3 Application of Routh-Hurwitz array:

\[ \text{OLTF} = \frac{K_C}{(s+1)(5s+1)(5s+1)} \] ....................................................... (4.5)

The characteristic equation:

\[ 1 + \text{OLTF} = 0 \]

\[ 1 + \frac{K_C}{(s+1)(5s+1)(5s+1)} \] ....................................................... (4.6)

\[ (s+1)(5s+1)(5s+1)+K_C = 0 \] ....................................................... (4.7)

The chr.eq:

\[ 25s^3 + 35s^2 + 11s + 1 + K_C = 0 \] ....................................................... (4.8)

Using Routh array:
This system is stable.

4.3.4 Using Direct Substitution Method:-

Obtain the frequency:

The Characteristic equation:-

\[25s^3 + 35 s^2 + 11 s + 1 + K_C = 0\] ..........................(4.8)

Set \(S= i\omega\), \((i^2 = -1)\):

\[-25 i\omega^3 - 35\omega^2 + 11 i\omega + 1 + K_C = 0\] ..........................(4.12)

Taking imaginary part of this loop=0
\[-25 \omega^3 + 11i\omega = 0 \] \hspace{1cm} \text{...(4.13)}

\[\omega^2 = 11\]

\[\omega = 0.6633 \text{rad/s}\]

Taking the real part of loop = 0

\[-35\omega^2 + 1 + K_C = 0 \] \hspace{1cm} \text{...(4.14)}

\[-35(0.6633)^2 + 1 + K_C = 0\]

\[K_C = K_u = 14.4\]

\[P_u = 2\pi/\omega_c\]

\[P_u = 2\pi / 0.6633 = 9.47 \text{sec}\]

\textbf{4.3.5 Apply the Root–Locus Using MATLAB:-}

\textbf{Plot Root–Locus:-}

\[\text{OLTF} = \frac{K_C}{(s+1)(s^2+1)(3s+1)} \] \hspace{1cm} \text{...(4.15)}

\textbf{MATLAB forner:-}

\[
\begin{align*}
& >> \text{num}=[1]; \\
& >> a=\text{conv([1 1],[5 1])}; \\
& >> \text{den}=\text{conv}(a,[5 1]); \\
& >> \text{sys}=\text{tf(num,den)} \\
\end{align*}
\]

\textbf{Transfer function:}

\[
\frac{1}{25s^3 + 35s^2 + 11s + 1}
\]

\[
>> \text{rlocus(sys)}
\]
\[ K_u = 14.4 \]

\[ \omega = 0.662 \text{ rad/sec} \]

\[ P_u = 2\pi/0.662 = 9.49 \text{ sec} \]

### 4.3.6 System Stability Using Bode Method:

Determination of ultimate period \((P_u)\) and ultimate gain \((K_u)\).

\[
\text{OLTF} = \frac{Kc}{(s+1)(s+1)(s+1)} \tag{4.16}
\]

MATLAB code:

```matlab
>> num=[1];
>> a=conv([1 1],[5 1]);
>> den=conv(a,[5 1]);
```
>> sys=tf(num,den)
Transfer function :
\[
\frac{1}{25s^3 + 35s^2 + 11s + 1}
\]
>> bode(sys),grid

Figure (4.18) Bode diagram for loop (2)

\[ \omega = 0.663 \text{rad/sec} \]

magnitude(dB) = -23.2

20\log_{10} AR = dB

20\log_{10} AR = -23.2

AR = 0.0691

\[ K_u = \frac{1}{AR} \]

\[ K_u = \frac{1}{0.0691} = 14.45 \]
\[ P_u = 2\pi/0.663 = 9.47 \text{sec} \]

### 4.3.7 Determination of adjustable parameters:

The average \( (P_{u,av}) = (P_{u,1} + P_{u,2} + P_{u,3}) / 3 = 9.48 \text{sec} \)

The average \( (K_{u,av}) = (K_{u,1} + K_{u,2} + K_{u,3} + K_{u,4}) / 4 = 14.4 \)

#### Table (4.6): Ziegler-Nichols adjustable controller parameters for additives loop for Routh-Hurwitz [23]

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>( K_c )</th>
<th>( \tau_i(min) )</th>
<th>( \tau_D(min) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>0.5Ku</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>0.45Ku</td>
<td>( \frac{pu}{1.2} )</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>0.6Ku</td>
<td>( \frac{pu}{2} )</td>
<td>( \frac{pu}{8} )</td>
</tr>
</tbody>
</table>

#### Table (4.7) Z-N Tuning parameters of loop 2

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>( K_c )</th>
<th>( \tau_i(min) )</th>
<th>( \tau_D(min) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>7.2</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>6.48</td>
<td>7.9</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>8.64</td>
<td>4.74</td>
<td>1.185</td>
</tr>
</tbody>
</table>

### 4.3.8 Response of the System for P Controller:

\[ Y_S = \pi_F / (1 + \pi_L) \]

\[ \pi_F = \frac{K_c}{(s+1)(5s+1)} \]

\[ 1 + \pi_L = 1 + \frac{K_c}{(s+1)(5s+1)(5s+1)} \]

\[ Y_S = \pi_F / (1 + \pi_L) \]
\[ Y_S = \frac{K_C(5s+1)}{25s^3+25s^2+11s^2+1+K_C} \] \hfill (4.84)

Putting \( K_C = 7.2 \)

\[ Y_S = \frac{36s+7.2}{25s^3+25s^2+11s^2+8.2} \] \hfill (4.85)

MATLAB form:

\begin{verbatim}
>> num=[36 7.2];
>> den=[25 35 11 8.2];
>> step(num,den),grid
\end{verbatim}

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure4.19.png}
\caption{Step Response Of loop 2 for P Controller.}
\end{figure}
Table (4.8) Simulation Parameter of Loop 2 for P Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time(sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>1.45</td>
</tr>
<tr>
<td>Beak time</td>
<td>4.34</td>
</tr>
<tr>
<td>Settling time</td>
<td>68.2</td>
</tr>
<tr>
<td>Over shoot</td>
<td>2.51</td>
</tr>
</tbody>
</table>

$\text{Decay Ratio } = \frac{1.55}{2.51} = 0.617$

4.3.9 System Response for PI-Controller:

From (Z-N) table for PI-Controller

$$K_c = 6.48 \quad \tau_f = 7.9$$

num=[51.192 6.48];
den=[7.9 0];
m=tf(num,den);
um1=1;
den1=[1 1];
r=tf(num1,den1);
um2=1;
den2=[5 1];
w=tf(num2,den2);
um3=1;
den3=[5 1];
x=tf(num3,den3)
z=feedback(m*r*w,x)
step(z)

Transfer function:

$$\frac{256s^2 + 83.59s + 6.48}{197.55^4 + 276.55^3 + 86.9s^2 + 59.09s + 6.48}$$
Figure (4.20) Step Response Of loop 2 for PI Controller.

Table (4.9) Simulation Parameter of Loop 2 for PI Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time(sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>1.22</td>
</tr>
<tr>
<td>Beak time</td>
<td>4.73</td>
</tr>
<tr>
<td>Settling time</td>
<td>148</td>
</tr>
<tr>
<td>Over shoot</td>
<td>3.02</td>
</tr>
</tbody>
</table>

Decay Ratio = \( \frac{2.39}{3.02} = 0.791 \)
4.3.10 System Response for PID-Controller:

From (Z-N) table for PID-Controller

\[ K_c = 8.64 \quad \tau_i = 4.74 \quad \tau_d = 1.185 \]

\[
\begin{align*}
\text{num} &= [48.53 \quad 40.95 \quad 8.64] ; \\
\text{den} &= [4.74 \quad 0] ; \\
\text{m} &= \text{tf}(\text{num}, \text{den}) ; \\
\text{num1} &= 1 ; \\
\text{num1} &= 1 ; \\
\text{num2} &= 1 ; \\
\text{den2} &= [5 \quad 1] ; \\
\text{w} &= \text{tf}(\text{num2}, \text{den2}) ; \\
\text{num3} &= 1 ; \\
\text{den3} &= [5 \quad 1] ; \\
\text{x} &= \text{tf}(\text{num3}, \text{den3}) \\
\text{z} &= \text{feedback}(\text{m} \ast \text{r} \ast \text{w}, \text{x}) \\
\text{step} (z)
\end{align*}
\]

\[
S^3 + 253.3 S^2 + 84.15 S + 8.64
\]

\[
118.55^4 + 165.95^3 + 100.75^2 + 45.695 + 8.64
\]

Figure (4.21) Step Response Of loop 2 for PI Controller.
Table (4.10) Simulation Parameter of Loop 2 for PI Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>0.437</td>
</tr>
<tr>
<td>Beak time</td>
<td>2.97</td>
</tr>
<tr>
<td>Settling time</td>
<td>29.5</td>
</tr>
<tr>
<td>Over shoot</td>
<td>2.87</td>
</tr>
</tbody>
</table>

Decay Ratio = \( \frac{1.35}{2.87} = 0.470 \)
4.3.11 Response of the System for P, PI and PID Controller:

```matlab
num=[36 7.2];
den=[25 35 11 8.2];
sys=tf(num, den);
step(num, den)
hold
num1=[256 83.59 6.48];
den1=[197.5 276.5 86.9 59.09 6.48];
sys1=tf(num1, den1);
step(num1, den1)
num2=[1 253.3 84.15 8.42];
den2=[118.5 165.9 100.7 45.69 8.64];
sys2=tf(num2, den2);
step(num2, den2)
```

Figure (4.22) Step Response Of loop 1 for P, PI and PID Controller
4.4 Control of Composition in the Multi-effect evaporator:

Figure (4.23) Physical diagram for composition control loop 3

4.4.1 Composition Control Loop Transfer Function:

Figure (4.24) General block diagram of loop 3 feedback control
4.4.2 Over all Transfer Function of Loop (3):

\[ G_C = K_C \] \hspace{2cm} (4.1)

\[ G_V = \frac{1}{(10S + 1)} \] \hspace{2cm} (4.2)

\[ G_P = \frac{1}{(S^2 + s + 1)} \] \hspace{2cm} (4.3)

\[ G_M = 1 \] \hspace{2cm} (4.4)

Figure (4.25) Conventional loop 3 feedback control

4.4.3 Application of Routh-Hurwitz array:

\[ \text{OLTF} = \frac{K_C}{(10S + 1)(S^2 + S + 1)} \] \hspace{2cm} (4.5)

The characteristic equation:

\[ 1 + \text{OLTF} = 0 \]

\[ 1 + \frac{K_C}{(10S + 1)(S^2 + S + 1)} \] \hspace{2cm} (4.6)
num = [1];

\[ \text{den} = \text{conv } ([10 1], [1 1 1]); \]

\[ \text{sys} = \text{tf}(\text{num}, \text{den}); \]

Transfer function:

\[
\frac{1}{10s^3 + 11s^2 + 11s + 1}
\]

\[(10s+1)(s^2+s+1)+K_C = 0 \] \hspace{1cm} (4.7)

The chr.eq:

\[10s^3 + 11 s^2 + 11 s + 1+ K_C = 0 \] \hspace{1cm} (4.8)

Using Routh array:

Number of rows = 4

\[
\begin{array}{ccc}
S^3 & 10 & 11 \\
S^2 & 11 & 1+K_C \\
S^1 & b_1 & b_2 \\
S^0 & c_1 & c_2 \\
\end{array}
\]

\[
\begin{array}{ccc}
S^3 & 10 & 11 \\
S^2 & 11 & 1+K_C \\
S^1 & (111-10K_C)/11 & 0 \\
S^0 & 1+K_C & 0 \\
\end{array}
\]

\[(111-10K_C)/11 = 0 \] \hspace{1cm} (4.9)

\[K_C = 11.1 \] \hspace{1cm} (4.10)
Ku=Kc = 11.1………………………………………………………………….. (4.11)

This system is stable.

4.4.4 Using Direct Substitution Method:-

Obtain the frequency:

The Characteristic equation:-

\[10s^3 + 11 s^2 + 11 s + 1 + Kc = 0\] ................................. (4.8)

Set S= i\omega , (i^2 = -1) :-

\[-10 i\omega^3 - 11\omega^2 + 11 i\omega + 1 + Kc = 0 \] ................................. (4.12)

Taking imaginary part of this loop=0

\[-10 i\omega^3 +11i\omega = 0 \] ................................. (4.13)

\[\omega^2 = 11/10 \]

\[\omega = 1.048\text{red/s} \]

Taking the real part of loop =0

\[-11\omega^2 +1 +Kc = 0 \] ................................. (4.14)

\[-11(1.048)^2 +1 +Kc = 0 \]

Kc = Ku= 11.1

P_u=2\pi/\omega_c0

P_u =2\pi / 1.048= 5.99sec

4.4.5 Apply the Root – Locus Using MATLAB:-

Plot Root – Locus:-

\[\text{OLTF}= \frac{Ke}{(10s+1)(s^2+s+1)} \] ................................. (4.15)
MATLAB former:

\[
\text{num}=[1]; \\
\text{den}=\text{conv}([10 1],[1 1 1]); \\
\text{sys}=$\text{tf(num,den)}$ \\
\text{Transfer function:} \\
\frac{1}{10.5^2+11.5^2+11.5+1} \\
\text{rlocus(sys)}
\]

Figure (4.26) Root Locus of loop (3)

\[K_u = 11.3\]

\[\omega = 1.06 \text{ rad/sec}\]
\[ P_u = \frac{2\pi}{1.06} = 5.92 \text{sec} \]

### 4.4.6 System Stability Using Bode Method:

Determination of ultimate period \((P_u)\) and ultimate gain \((K_u)\).

\[
\text{OLTF} = \frac{Kc}{(10s+1)(s^2+s+1)} \tag{4.16}
\]

MATLAB former:

```
num=[1];
den=conv([10 1],[1 1 1]);
sys=tf(num,den);
```

Transfer function:

\[
\frac{1}{10s^3+11s^2+11s+1}
\]

```
bode(sys),grid
```

![Bode Diagram](image)
Figure (4.27) Bode diagram for loop (3)

\[ \omega = 1.04 \text{rad/sec} \]

Magnitude (dB) = -21.1

\[ 20 \log_{10} AR = \text{dB} \]

\[ 20 \log_{10} AR = -21.1 \]

\[ AR = 0.088 \]

\[ K_u = \frac{1}{AR} \]

\[ K_u = \frac{1}{0.088} = 11.3 \]

\[ P_u = \frac{2\pi}{1.04} = 6.04 \text{sec} \]

4.4.7 Determination of adjustable parameters:

The average (\( P_{u,av} \)) = (\( P_{u,1} + P_{u,2} + P_{u,3} \)) / 3 = 5.98 sec

The average (\( K_{u,av} \)) = (\( K_{u,1} + K_{u,2} + K_{u,3} + K_{u,4} \)) / 4 = 11.2

Table (4.11): Ziegler-Nichols adjustable controller parameters for additives loop for Routh -Hurwitz [23]

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>Kc</th>
<th>( \tau_1 (\text{min}) )</th>
<th>( \tau_D (\text{min}) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>0.5Ku</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI</td>
<td>0.45Ku</td>
<td>( \frac{pu}{1.2} )</td>
<td>-</td>
</tr>
<tr>
<td>PID</td>
<td>0.6Ku</td>
<td>( \frac{pu}{2} )</td>
<td>( \frac{pu}{8} )</td>
</tr>
</tbody>
</table>
Table (4.12) Z-N tuning parameters of loop 3:-

<table>
<thead>
<tr>
<th>Type of controller</th>
<th>$K_c$</th>
<th>$\tau_i(min)$</th>
<th>$\tau_d(min)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>5.6</td>
<td>_</td>
<td>_</td>
</tr>
<tr>
<td>PI</td>
<td>5.04</td>
<td>4.98</td>
<td>_</td>
</tr>
<tr>
<td>PID</td>
<td>6.72</td>
<td>2.99</td>
<td>0.747</td>
</tr>
</tbody>
</table>

4.4.8 Response of the System for P Controller:-

$$Y_S = \pi_F / (1 + \pi_L)$$

$$\pi_F = \frac{K_c}{(10s+1)(s^2+s+1)}$$  \hspace{1cm} (4.17)

$$1 + \pi_L = 1 + \frac{K_c}{(10s+1)(s^2+s+1)}$$  \hspace{1cm} (4.18)

$$Y_S = \frac{K_c}{10s^3+11s^2+11s+1+K_c}$$  \hspace{1cm} (4.19)

Putting $K_c = 5.6$

$$Y_S = \frac{5.6}{10s^3+11s^2+11s+5.6}$$  \hspace{1cm} (4.20)

MATLAB former:-

```matlab
num=[5.6];

den=[10 11 11 6.6];

step(num,den),grid
```
Figure (4.28) Step Response Of loop 3 for P Controller.

Table (4.13) Simulation Parameter of Loop 3 for P Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>3.16</td>
</tr>
<tr>
<td>Beak time</td>
<td>4.58</td>
</tr>
<tr>
<td>Settling time</td>
<td>26.6</td>
</tr>
<tr>
<td>Over shoot</td>
<td>1.12</td>
</tr>
</tbody>
</table>

Decay Ratio = $\frac{0.842}{1.12} = 0.84$
4.4.9 Response of the System for PI Controller:-

From (Z-N) table for PI-Controller

\[ K_c = 5.04 \quad \tau_i = 4.98 \]

\[ \text{num} = [25.09 \ 5.04]; \]
\[ \text{den} = [4.98 \ 0]; \]
\[ m = \text{tf}(\text{num}, \text{den}); \]
\[ \text{num1} = 1; \]
\[ \text{den1} = [10 \ 1]; \]
\[ r = \text{tf}(\text{num1}, \text{den1}); \]
\[ \text{num2} = 1; \]
\[ \text{den2} = [1 \ 1 \ 1]; \]
\[ w = \text{tf}(\text{num2}, \text{den2}); \]
\[ x = 1; \]
\[ z = \text{feedback}(m \ast r \ast w, x) \]
\[ \text{step}(z) \]

![Step Response Graph](image-url)

**Figure (4.29)** Step Response Of loop 3 for PI Controller.
Table (4.14) Simulation Parameter of Loop 3 for PI Controller:-

<table>
<thead>
<tr>
<th>Items</th>
<th>Time(sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>1.93</td>
</tr>
<tr>
<td>Beak time</td>
<td>5.22</td>
</tr>
<tr>
<td>Settling time</td>
<td>21.5</td>
</tr>
<tr>
<td>Over shoot</td>
<td>1.44</td>
</tr>
</tbody>
</table>

Decay Ratio = $\frac{1.13}{1.44} = 0.784$
4.4.10 System Response for PID-Controller:-

From (Z-N) table for PID-Controller

\[ K_c = 6.72 \quad \tau = 2.99 \quad \tau_d = 0.747 \]

\[
\text{num} = [15.009, 20.09, 6.72]; \\
\text{den} = [2.99, 0]; \\
m = \text{tf(num, den)}; \\
\text{num1} = 1; \\
\text{den1} = [10, 1]; \\
r = \text{tf(num1, den1)}; \\
\text{num2} = 1; \\
\text{den2} = [1, 1, 1]; \\
w = \text{tf(num2, den2)}; \\
x = 1 \\
z = \text{feedback(m*r*w, x)} \\
\text{step}(z)
\]

![Step Response Graph](image)

Figure (4.30) Step Response Of loop 3 for PID Controller.
Table (4.15) Simulation Parameter of Loop 3 for PID Controller:

<table>
<thead>
<tr>
<th>Items</th>
<th>Time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rise time</td>
<td>1.58</td>
</tr>
<tr>
<td>Break time</td>
<td>3.9</td>
</tr>
<tr>
<td>Settling time</td>
<td>14.1</td>
</tr>
<tr>
<td>Over shoot</td>
<td>1.37</td>
</tr>
</tbody>
</table>

Decay Ratio $= \frac{1.08}{1.37} = 0.788$

4.4.11 Response of the System for P, PI and PID Controller:

```matlab
num=[5.6];
den=[10 11 11 6.6];
sys=tf(num,den);
step(num,den)
hold
num1=[25.09 5.04];
den1=[49.8 54.78 54.78 30.07 5.04];
sys1=tf(num1,den1);
step(num1,den1)
num2=[15.01 20.09 6.72];
den2=[29.9 32.89 47.9 23.08 6.72];
sys3=tf(num2,den2);
step(num2,den2)
```
Blue=P, Green=PI, Red=PID

Figure (4.31) Step Response Of loop 1 for P, PI and PID Controller.
4.5 Discussion:-

The control of the mixer and Multi Effect Evaporators system used four methods: Routh Hurwitz method, direct substitution, Root Locus and Bode method.

These methods are proved to be identical in the results and each of them is qualified to be used for tuning and stability analysis without loss of accuracy.

After using the three types of controller (P, PI, and PID) to control each of the mixer composition, the third evaporator pressure and the composition of the product; we found that the (PID) controller has the fastest response after comparing between them.
CHAPTER FIVE

Conclusions and
Recommendations
Chapter Five

Conclusions and Recommendations

5.1 Conclusions:-

1. It was concluded that the selecting of an effective tuning method depend on the highest gain and the best performance of the methods, in this research four methods were used, it was found that all methods were effective for pressure control loop and composition control loop which give the highest gain in each control loops.

2. Three loops were used to control the system pressure and composition.

3. A control system was designed, tuned and checked for stability.

5.2 Recommendations:-

Based on the study we recommend the following:-

1. A feedback control loop should be applied on the fourth evaporator to control the pressure and the composition.

2. A feedback control loop should be applied on the mixer to control the composition.

3. The process control system should be applied practically in any continuous chemical process systems.

4. The process control system should be applied for the production of caustic soda in the form of crystals so that it could be easy to transport from one place to another.

5. A control system should be designed for the electrolysis cell to control the temperature, pressure and composition of the cell.

6. The study recommended using Cohen-Coon tuning method and comparing it with Ziegler-Nichols.
5.3 References: