SOFT SENSING AND CONTROL OF PRODUCT COMPOSITION OF REACTIVE DISTILLATION COLUMN USING DYNAMIC NEURAL NETWORK

Ph.D. Thesis

GAURAV KATARIA

ID No. 2014RCH9514

DEPARTMENT OF CHEMICAL ENGINEERING

MALAVIYA NATIONAL INSTITUTE OF TECHNOLOGY JAIPUR

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Soft Sensing and Control of Product Composition of Reactive Distillation Column Using Dynamic Neural Network

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GAURAV KATARIA

ID No. 2014RCH9514

Under the Supervision of

Prof. Kailash Singh

DEPARTMENT OF CHEMICAL ENGINEERING

MALAVIYA NATIONAL INSTITUTE OF TECHNOLOGY JAIPUR

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DECLARATION

I, **Gaurav Kataria**, declare that this thesis entitled **"SOFT SENSING AND CONTROL OF PRODUCT COMPOSITION OF REACTIVE DISTILLATION COLUMN USING DYNAMIC NEURAL NETWORK"** and the work presented in it, are my own. I confirm that:

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CERTIFICATE

This is to certify that the thesis entitled **"SOFT SENSING AND CONTROL OF PRODUCT COMPOSITION OF REACTIVE DISTILLATION COLUMN USING DYNAMIC NEURAL NETWORK"** being submitted by **Gaurav Kataria (2014 RCH9514)** is a bonafide research work carried out under my supervision and guidance in fulfillment of the requirement for the award of the degree of **Doctor of Philosophy** in the Department of Chemical Engineering, Malaviya National Institute of Technology Jaipur, India. The matter embodied in this thesis is original and has not been submitted to any other University or Institute for the award of any other degree.

Place: Jaipur

Date:

Kailash Singh
Professor
Dept. of Chemical Engineering
MNIT Jaipur

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ABSTRACT

The real time control of a chemical unit is an essential factor which can save a lot of money and can lead to high performance of a plant. To control any chemical plant in a real time, the measuring element has to send on-line estimation signals leading to the use of soft sensor. The reactive distillation column (RDC) is a complex nonlinear plant which carries two processes: reaction and separation at the same time. This complexity causes the RDC to be a difficultly controlled chemical process equipment. Since a reactor and a distillation column performance can be signified by its product composition instead of reactor temperature or tray temperature, the direct estimation and control of product composition is a preferable scheme to control the RDC unit. This product composition is difficult to measure online using a hardware sensor because of the presence of dead time in the sensor, leading to the demand of an online composition estimator for an RDC.

For the real time monitoring of an RDC, a Recurrent Neural Network (RNN) based soft sensor has been proposed to estimate the bottoms product composition of an RDC for the synthesis of *n*-Butyl Acetate using esterification reaction. When the sensor composition and true composition were compared, it was observed that the maximum mean square error (MSE) was obtained in the order of 10^{-6}. This soft sensor acts as a measuring element in a closed loop involving a PI controller for the direct control of the RDC product composition. During the study of open loop response of RDC for various step changes, it was observed that during the step change in acetic acid feed rate, the RDC model was showing an inverse response. To deal with this inverse response problem due to step change in a load variable, a feedforward-feedback controller was developed to control the RDC. The controller performance was compared with a closed loop having feedback controller only. During the study of inverse response, it was observed that the problem of inverse response due to manipulating variable has only been solved for a 2^{nd} order or reduced 2^{nd} order transfer function models by other researchers. This study also involves work on an inverse response compensator for a 3^{rd} order transfer function model. The control system performance for the process was compared in the presence and absence of proposed compensator in the closed loop.

When the feedforward-feedback controller was applied to the RDC mathematical S-function model, RDC non-linearity worsened the controller performance for an increase in acetic acid feed rate. Therefore some other controller was required to deal with problem of inverse response due to load variable that works on future prediction of controlled variable. This future prediction characteristics was expected to cure the control problem in the plant by deciding the process input accordingly when a controller sees an inverse response in the future and a Model Predictive Control (MPC) is proposed for the study. Since the transfer function model was unable to include all the non-linearities of the RDC, a dynamic neural network model was used inside the MPC which encloses all the non-linearities of RDC. The closed loop performance of RDC for set point changes and disturbance rejection was evaluated during the presence and absence of dead time in the RDC. In this study, the performance of PI, feedforward-feedback controller and MPC has been evaluated and compared using performance indexes such as integral of square of errors (ISE), integral of absolute errors (IAE), and integral of time-weighted absolute errors (ITAE). The integral errors decreased in the range from 10% to more than 100% of the error value while using the proposed control techniques.

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Publications from the Thesis

Published in Journal

1. Kataria G. and Singh K., "Recurrent Neural Network based Soft Sensor for Monitoring and Controlling a Reactive Distillation Column" Chemical Product and Process Modeling, 13(3), 2018.

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2. Kataria G. and Singh K., "Removing Inverse Response Due to Load Variable using Feedforward-feedback Controller", An IEEE proceeding of the 4^{th} Indian control conference (ICC 2018), IIT Kanpur, 4-6 January, 2018.

3. Kataria G. and Singh K., "Comparison of Levenberg-Marquardt and Gradient Descent Based Soft Sensors for Reactive Distillation Column", CHEMCON 2017, Anna University, Chennai, 27-30 December, 2017.

4. Kataria G. and Singh K., "RNN Based Soft Sensor for Estimation of Concentration for a Reactive Distillation Column", An IEEE proceeding of the National Conference on Process Automation & Control (NCPAC 2016), MNNIT Allahabad, 28-30 May, 2016.

5. Kataria G., Singh K. and Dohare R. K. , "ANN Based Soft Sensor Model for Reactive Distillation Column", National Conference on Process Automation & Control (NCPAC), 28-29 May 2016, MNIT, Jaipur

6. Kataria G. and Singh K., "Optimization of Product Purity in Reactive Distillation Column", CHEMCON 2015, IIT Guwahati, 27-30 December, 2015.
7. Kataria G. and Singh K., "Dynamic model based feedforward control of a reactive distillation column for synthesis of butyl acetate", CHEMCON 2018, NIT, Jalandhar, 27-30 December, 2018. (Accepted)

Chapter 1

Introduction

1.1 Introduction

Real-time prediction of the quality variables has become a crucial step in maintaining the performance of the process. Getting strict on pollution standards, an increase of competition in the market, and maintaining the quality of the product has been a big inspiration to develop an on-line estimator for the estimation of quality variables. But this estimator should be reliable and cheap enough so that it does not add an extra cost to the plant. Since many hardware sensors are costly to maintain and carry some transport delay in their processing, they are unable to estimate the variable online. It heads to the development of the soft sensor for the on-line estimation of quality variables.

A soft sensor or inferential estimator is a mathematical model which is used to estimate the difficultly measured variables or primary variables using easily measured variables or secondary variables. In a distillation column, the distillate and bottom product compositions are the primary variables and tray temperatures are the secondary variables for the sensor. The estimation of the product composition is carried out using an equipment gas chromatography(GC). The GC is an expensive and high maintenance technique and cannot help in online estimation because of the presence of some time lag in the measurements. Since the tray temperatures can easily be measured on-line using thermocouples, a soft sensor can be a fast and cheap alternative which can measure the product composition using tray temperatures of the column. A soft sensor application lies in the chemical plants having distillation columns, reactors, air separators, etc.

A reactive distillation (RD) is a process that involves both reaction and separation in a single column. The reversible reactions are preferred in an Reactive Distillation Column (RDC) so that backward reaction can be restricted in the process. The prime motive of an RDC is to separate the products of the reaction simultaneously as they are being produced so that Le Chatelier's principle can restrain the backward reaction. The RDC holds the advantages of better selectivity, better conversion, avoidance of azeotropes formation and better heat utilization. The esterification reaction is a reversible reaction which has been studied extensively in the RDC. The esterification involves the formation of an ester from the reaction of carboxylic acid and alcohol in the presence of an acidic catalyst. The product composition is a prime indicator of an RDC performance, its on-line monitoring is essential for the tighter control. This leads to the use of soft sensors for an RDC.

Soft sensors are generally developed using principal component analysis(PCA) (Zamprogna et al., 2005; Venkateswarlu and Jeevan Kumar, 2006; Qiao et al., 2010; Canete et al., 2012), partial least square(PLS) (Fujiwara et al., 2012a; Mohamed Ramli et al., 2014; Shang et al., 2014; Kaneko and Funatsu, 2013) , artificial neural network(ANN) (Bahar and Özgen, 2010; Khazraee and Jahanmiri, 2010; Rogina et al., 2011; Vijaya Raghavan et al., 2011; Zhou et al., 2012; Canete et al., 2012; Shang et al., 2014) , and support vector regression(SVR)Qiao et al. (2010); Gholami and Shahbazian (2015); Behnasr and Jazayeri-Rad (2015) . Among these techniques, PCA and PLS are the linear models whereas ANN and SVR are nonlinear in nature. In the case of neural networks, Recurrent Neural Network (RNN) is a nonlinear dynamic neural network technique whose current output depends on current input as well as previous output of the process. This characteristic of RNN makes the network to be dynamic as well as adaptive upto some extent. The RNN technique has been used to develop a soft sensor for the real time estimation of butyl acetate composition in bottoms stream of RDC using tray temperatures of the column.

While studying the open loop response of the RDC model discussed in Chapter 3, it was observed that RDC is showing an inverse response. The inverse response happens when two opposing processes works simultaneously and have different time constants and gains. The inverse response causes the model response to change the direction in the end of the process as it was opted by the system during the start of the step change. In the case of RDC, the inverse response has been observed during the step change in acetic acid feed rate causing sluggishness and higher overshoots in the closed loops. Since this inverse response is due to the step change in load variable instead of manipulating variable, a conventional compensator cannot be designed for the problem. To deal with the problem of inverse response due to load variable the controller needs to have feedforward characteristics along with feedback controller to restrict the disturbance entrance into the process so that it cannot add inverse response in the system.

The literature work on inverse response shows that the problem is handled only for a 2^{nd} order or reduced 2^{nd} order transfer function process models but not for third order systems. It has been mentioned in Iinoya and Altpeter (1962) and Stephanopoulos (1984) that a third order process can also have a problem of inverse response due to step change in manipulating variable. So the study can also be extended to design a compensator for the problem of inverse response due to manipulating variable in a 3^{rd} transfer function model.

The problem of inverse response due to load variable can also be handled using a controller with future prediction characteristics. The future predictions can help the controller to optimize the plant input accordingly by identifying that inverse response is going to held in the future. The model predictive control (MPC) is an advanced control technique which optimizes the plant input using the future model prediction data for a confined horizon. It has been discussed in Perry and Green (2008) that MPC can easily handle the problem of dead time and inverse response using its future predictions factor. Since MPC depend on the inside model, the model has to be accurate enough that controller can rely on its future predictions. An RDC is a complex, nonlinear and a dynamic plant whose model has to be accurate enough to cover all the non-linearity and dynamic properties of the RDC. To deal with these properties a neural network is a better technique which can cover RDC nonlinear properties at all the input and output ranges. To cover the dynamic properties of the RDC, a dynamic neural network can be used as an internal MPC model which deal with dynamic sequential input-output data of an RDC.

1.2 Research Gaps

Based on the literature survey discussed in Chapter 2, the following gaps were identified for the study.

1. Composition is a direct measure of an RDC's performance, and it should be monitored online. The techniques like gas chromatography (GC) measure the compo-

sition directly but with some time lag making the GC to be unsuitable for online monitoring. There is a need to develop an estimator for an RDC, which can give online estimation without any time lag. As RDC is a complex nonlinear model which carries both separation and reaction in a single still, a soft sensor for RDC has to be compatible with its non-linearity and complexity.

2. The alcohols studied in RDC for the formation of an ester are methanol, ethanol, isopropanol, n-butanol, and n-pentanol. The soft sensing technique has only been applied to the production of ethyl ester, leaving a broad range of the reaction systems to study.

3. Chemical plants are highly non-linear in nature and go through a lot of variations with the time. The changes in a plant can be handled using adaptive techniques of soft sensors. Many adaptive soft sensors have already been proposed for a distillation column (Rani et al., 2013; Shang et al., 2014; Shao et al., 2015; Kaneko and Funatsu, 2015). For reactive distillation columns, Khazraee et al. (2010) and Vijaya Raghavan et al. (2011) developed an adaptive sensor by using Adaptive Neuro-Fuzzy Inference System(ANFIS) technique. Besides ANFIS, no other adaptive soft sensor technique has been applied on RDC leaving a wide range of adaptive advanced soft sensing techniques to study for reactive distillation columns. The RNN technique has not been applied to the best of our knowledge.

4. In the case of neural network techniques, networks having both dynamic and adaptive characteristics are still needed to be explored for the soft sensing purpose.

5. In the case of problem of Inverse Response due to load variable, literature shows no solution for this.

6. In the case of Inverse Response due to manipulated variable, compensators were designed by transforming the system into a 2^{nd} order or reduced 2^{nd} order transfer function model. A compensator for the 3^{rd} order systems has not been explored.

7. The soft sensors are generally linked with the PID controllers instead of advanced controllers. Coupling of advance neural network techniques for both the sensor

and controller is not explored much in literature.

8. The combination of dynamic sensor and controller is not explored much in the literature, and combination of RNN based soft sensor and Dynamic Neural Network (DNN) based MPC has not been explored yet.

1.3 Research Objectives

Considering the above mentioned gaps, the following objectives were outlined for the research study.

1. To develop a mathematical dynamic model of a reactive distillation column and validate the model with any experimental study present in the literature.

2. To develop a recurrent neural network based soft sensor which works on the input values of tray temperatures and produce the bottoms composition of butyl acetate.

3. To develop a control system by using an RNN based soft sensor as a measuring element in the closed loop.

4. To design a feedforward feedback controller to minimize the effect of inverse response due to the load variable.

5. To develop a solution of curing the problem of an inverse response due to manipulated variable for a 3^{rd} order transfer function model.

6. To develop a dynamic neural network model of an RDC with a good prediction accuracy.

7. To develop a dynamic neural network based model predictive controller for solving the problem of an inverse response and controlling the RDC with better performance.

1.4 Thesis Overview

The thesis is further distributed into the following chapters.

1. The Chapter 2 discusses the previous work on developing a soft sensor, developing a soft sensor for a reactive distillation column, handling of inverse response problem and developing a model predictive controller using various techniques.

2. In Chapter 3, a brief overview of reactive distillation column is presented. The mathematical model of RDC is formulated along with its validation and an open loop study of the model at different step changes in the load variables.

3. The Chapter 4 discusses about the problem of inverse response due to both manipulating and load variable. The solution for both the problems is presented.

4. The Chapter 5 discusses the algorithm of recurrent neural network which has been used to develop a soft sensor model to estimate the product composition of RDC. The performance of soft sensor is analyzed at both the open loop and closed loop responses of the model.

5. In Chapter 6, the model predictive control technique has been covered whose internal model is based on dynamic neural network technique. The dynamic neural network based MPC has been used to control the product composition of RDC at both servo and regulatory response system when model is with and without transport delay.

6. The Chapter 7 discusses the overall conclusion of the study and work which can be explored in future by extending the study.

Chapter 2

Literature Survey

Before proceeding to the work on the soft sensor, inverse response and model predictive control (MPC), the study of previous work in the literature is necessary. The literature will give the insights of the previous work that has been already done and gaps which needs to be filled out with our study. This chapter includes literature survey on developing the soft sensor models, handling the inverse response problems and techniques and the applications of model predictive control.

2.1 Development of Soft Sensor Model

A soft sensor is an inferential estimator which estimates the quality variables using other secondary variables. The soft sensor has been used for estimating various quality measurement variables for several distillation columns, reactors and other chemical engineering units. This section will show some of the literature survey involving different development techniques and applications of the soft sensors.

Rao et al. (1993) developed an intelligent soft sensor to estimate kappa number and cooking time for a batch digester in a pulp mill. In the intelligent soft sensor, output variables of the sensor depends upon the quality of the input variables along with their quantity. The quality of the input variables helps the operator to decide the operating conditions for the process. Results showed that the intelligent soft sensor proved to be a good estimator for estimating the value of kappa number.

Dong et al. (1995) extended the linear techniques of partial least square (PLS) and principal component analysis (PCA) to non-linear counterparts. They developed a soft sensor based on the neural network partial least square (NNPLS) technique to estimate the composition of NO_x gas in exhaust stream for the industrial heater. They also used non-linear principal component analysis (NLPCA) for the sensor input data analysis. In the data analysis, all the less important input variables were refined from the raw data and then passed as the sensor input data. NNPLS based soft sensor gave better and robust results than linear PLS technique.

Wang et al. (1996) had developed a soft sensor for case-based modeling of the systems. High purity distillation column was studied for the case-based modeling, and a soft sensor model was developed to estimate the distillate and bottom product composition. Fuzzy distributed radial basis function (RBF) neural network technique was used to develop a soft sensor that solved the problem of relating highly non-linear input-output case data and gave promising results of the output.

The soft sensor is generally used to estimate one to two quality variables but Dayal and MacGregor (1997) implemented soft sensor for estimating ten primary variables including flow rate, lead content, etc. in industrial mineral flotation circuit. They used recursive weighted partial least square (PLS) technique to develop the soft sensor. The results showed that soft sensor predicted the outputs with significantly small mean square error (MSE). The method also holds the benefit of handling the problem of correlated variables.

Kalman filter is an old linear technique used for developing linear soft sensors. Baratti et al. (1998) considered widening Kalman Filter technique to Extended Kalman Filter (EKF) for the sensing purpose. Extended Kalman Filter is the nonlinear counterpart of Kalman filter technique. They developed EKF based soft sensor to estimate the distillate stream product composition for a distillation column. The results showed that experimental and sensor output values were adequately similar to each other.

Hong et al. (1999) developed the soft sensor based on neural network partial least square (NLPLS) technique for estimating the distillate product composition of a simulated binary distillation column and industrial splitter column. The soft sensor proposed by them also includes denoising of the signals and weighting of the variables. Results showed that NLPLS is superior method than linear PLS.

Frattini Fileti et al. (1999) integrated the soft sensor with self tuning controller for the multicomponent batch distillation column. A soft sensor was developed using artificial neural network (ANN) technique for the estimation of distillate composition. When gas chromatography (GC) and soft sensor results were compared, it was observed that ANN based soft sensor was compatible with online monitoring of plant and can handle online operating problems of a real plant.

Park and Han (2000) proposed a locally weighted regression (LWR) technique to develop a soft sensor for the estimation of bottom product composition for an industrial splitter column and a temperature of the 90% distillate diesel for the industrial crude column. The LWR technique was a simple non-linear method for handling the col-linearity problem of the input variables and the non-linearity problem of the system. They observed that LWR based soft sensor outperformed PLS, nonlinear PLS and ANN based soft sensors.

Partial least square (PLS) regression is a popular linear technique for the development of a soft sensor. Zamprogna et al. (2002) stretched the study of conventional PLS to multiple PLS. They developed a soft sensor based on the multiple PLS method to estimate the distillate composition of a batch distillation column. In multiple PLS, batch data was subdivided into subsets where each subset corresponds to different operating period. The input-output data was regressed using different multiple PLS techniques such as linear, polynomial, spline and ANN PLS. Results pointed that multiple PLS is better than conventional PLS. Among all the multiple PLS models, linear and polynomial models gave the best results with least error during prediction of quality variable.

Zamprogna et al. (2004) developed a soft sensor based on multiple partial least square (MPLS) combined with principal component analysis (PCA). The role of PCA was to analyze the input data for solving the multicollinearity problem. It was observed that MPLS was computationally less expensive technique for the soft sensing purpose than neural network techniques.

Opting all the tray temperatures or other variables as input variables can cause multicollinearity problem in the soft sensor. Zamprogna et al. (2005) suggested the use of principal component analysis (PCA) technique for refining input variables to cope up with multicollinearity obstacle. They firstly used PCA method for sensitivity analysis and then developed the soft sensors using partial least square PLS, Nonlinear PLS (NPLS) and artificial neural network (ANN) techniques. Promising results were obtained with all the soft sensors when inputs were reduced using PCA method.

Fortuna et al. (2005) presented a soft sensor based on neural network for predicting the top and bottom product compositions of a debutanizer column. They compared the results of soft sensor with real plant data and observed that neural network based soft sensor can handle the non linear processes and can deal with real-time process data.

Data preprocessing is considered as a significant step in soft sensor development, the presence of outliers and missing values in the input data can cause some reduction in the efficiency of a soft sensor. To handle the above problem Lin et al. (2007) proposed Univariate Hampler Identifier and Multivariate principal component analysis technique to manage the input data. They developed a dynamic partial least square (DPLS) based soft sensor for the prediction of free lime and NO_x gas composition in a cement kiln system. Above techniques showed promising results and solved the problem of outliers and missing data and also handled numerically large variance values.

Earlier for nonlinear processes, soft sensing was majorly performed using ANN technique, but ANN drawback was that it can get stuck into a local minima. To solve the above problem, Jain et al. (2007) proposed a soft sensor based on the support vector regression (SVR) which only search for the global minima. SVR based soft sensor was developed to estimate the product composition of batch distillation column and the results obtained were very close to the actual values solving problem of local minima.

Yan (2008) proposed a soft sensor based on the modified nonlinear generalised ridge regression (MNGRR) for the estimation of Naphtha 95% cut point in a crude distillation column. MNGRR solves the problem of multicollinearity in the input variables. It required less parametric sensitivity than other methods, and it also obtains the global optimum solution for the problem. Results were produced with small error which was very promising for the system.

LI et al. (2009) estimated light naphtha end point in a hydrocracker fractionator unit using a soft sensor based on the Affinity Propagation (AP) Clustering with Gaussian Process Regression (GPR) and Bayesian Committee Machine (BCM). They clustered the input variables according to their operating points using AP technique. Then GPR was used to obtain sub-models estimation and lastly, BCM was implemented to get a global probabilistic prediction. The techniques mentioned above does not require any pre-decided number of clusters in the system and also provide helpful probabilistic non-parametric model.

Ma et al. (2009) developed a soft sensor based on step-wise linear regression. They estimated the isopropylbenzene impurity in the distillate stream of O-xylene distillation column using the soft sensor. The square root filter was used to improve the numeric features of the algorithm. The step-wise linear regression method was useful for the local linear models showing adaptive behavior with less problem of multicollinearity.

Qiao et al. (2010) used a principal component analysis (PCA) technique with least square support vector regression (LS-SVR) to develop a soft sensor model for estimating free calcium oxide (f-CaO) contents in a cement clinker calcination process. They also considered detection and removal of outliers as an important step for the soft sensing. The results explained that implementing soft sensor on cement clinker calcination plant is a successful approach for the real-time estimation and control of a cement plant.

Galicia et al. (2011) discussed a dynamic extension of the partial least square technique. They developed soft sensor using dynamic PLS (DPLS) and reduced order DPLS (RO-DPLS) techniques for the estimation of kappa number in a simulated single-vessel digester and an industrial Kamyr digester. The DPLS technique holds a disadvantage that its theoretical knowledge is not well defined leading to the confusion about its capability to capture the dynamics of process model adequately. The technique also does not provide fair estimation when closed loop procedure has not been studied rigorously. But the above drawbacks of DPLS were solved using RO-DPLS when it was applied to the system and gave better results than DPLS.

To make a soft sensor adaptive in nature; model reconstruction is a significant but time and memory consuming step. Kaneko and Funatsu (2011) developed a partial least square (PLS) Time difference (TD) based soft sensor which overcomes the above problem of model reconstruction. They made an adaptive PLS-TD based soft sensor to estimate the bottom product composition in a crude distillation column. The results obtained had good accuracy and does not had any drawback of time and memory high consumption.

Rogina et al. (2011) proposed a soft sensor based on the multiple layer perceptron (MLP) and radial basis function (RBF) neural networks to estimate the light naphtha vapor pressure in a crude distillation column. The prime objective was to deal with the highly nonlinear process model. Results showed that neither multiple linear regression (MLR) nor artificial neural network (ANN) based soft sensors were that promising than MPL and RBF neural networks based soft sensors for dealing with highly nonlinear models.

Fujiwara et al. (2012a) presented a soft sensor to estimate the ethylene composition in an ethylene fractionator using PLS with Nearest Correlation Spectral Clustering Variable Selection (NCSC-VS) technique. The role of NCSC method is to preprocess the input data. The NCSC discards the irrelevant input variables by organizing them into the clusters of variables with different classes. Each class corresponds to its particular contribution to the output. When results were compared with other parameter selection techniques, NCSC-VS proved to have maximum accuracy than other methods.

Canete et al. (2012) developed a soft sensor and used it as a measuring element in a control system. They developed a soft sensor from adaptive neural network technique and used the principal component analysis (PCA) technique for the selection of input variables. They dealt with the ethanol-water distillation column and developed a soft sensor to estimate its distillate and bottom product composition. They observed that a quick response can be experienced when set point and null stationary errors are changed gradually.

Galicia et al. (2011) compared reduced order dynamic partial least square (RO-DPLS) technique with dynamic PLS (DPLS). While Galicia et al. (2012) extended their comparative studies by comparing the performance of RO-DPLS linked with different updating schemes like recursive PLS (RPLS), block PLS (BPLS), moving window PLS (MWPLS) and forgetting factor PLS (FFPLS). They developed the soft sensors based on all the above techniques for estimating kappa number for a pulp digester. Results revealed that RO-DPLS, when associated with RPLS, gave better results than all the other methods.

Zhou et al. (2012) proposed a soft sensor for estimating the Kerosene dry point for an atmospheric distillation column using a technique of bootstrap based neural network and bootstrap based partial least square. The bootstrap method leads to more reliability and robustness of the model. In bootstrap neural networks, multiple networks are taken and combined to get a better outcome. Results showed that bootstrap neural network and PLS proved to be better techniques than simple ANN and PLS and also solved the problem of over-fitting and worked fine with small input data set.

The performance of a soft sensor is significantly affected because of any missing data in the input data sets. Jin et al. (2012) developed a soft sensor to overcome this problem when missing data is persistent in the system. They developed soft sensor to estimate the biomass composition in a fermentation reactor using linear parameter varying (LPV) with expectation maximization (EM). The purpose of EM was to handle the missing data and LPV was used to manage the non-linearity in the system. The results explained that above given techniques gave better results than regular missing data handling methods. Canete and Saz-orozco (2012) used neural network based methodology for the identification and control of an ethanol-water distillation column. It was observed that above technique requires less parameters than other techniques. When estimated and actual values were compared, results showed that neural network based soft sensor gave quite an accurate results. The neural network based controller was also proved to be better than a PID controller.

Without input data preprocessing, the less useful data deteriorates the efficiency of the soft sensor. Wang et al. (2013) developed a soft sensor based on partial least square with non-negative garrote (PLSNNG) technique to make a sensor free from insubstantial overloaded input variables. The work of non-negative garrote was to compress the input data into few latent variables and remove all the other unnecessary variables throughout the running time of the process. When the results of PLS and PLSNNG were compared, PLSNNG revealed more accurate results than PLS method.

Fujiwara et al. (2012b) developed a soft sensor based on partial least square technique linked with nearest correlation (NC)- correlation based Just in time (CoJIT) modeling. A process plant goes through many process characteristics changes and individual differences can be observed in production devices. The work of NC technique is to cope up with individual differences of the production devices and CoJIT is used to cope up with changes in process characteristics. The results showed that measured and estimated values are very much similar to each other. Thus depicting CoJIT based PLS soft sensor is a suitable sensor for the long time running processes.

Rani et al. (2013) proposed the adaptive neural network technique for adapting the changes in the input variables. They developed the soft sensors based on Levenberg-Marquardt (LM) and dynamic adaptive linear network (D-ADALINE) for estimating top and bottom product composition for the multicomponent distillation column. They connected both soft sensors with ADALINE and D-ADALINE based inferential controllers in a control system. When results were compared, it was observed that D-ADALINE based soft sensor linked with D-ADALINE based controller proved to be the best combination for the system and gave the most accurate results.

Soft sensors are mainly applied to various distillation columns, but other complex processes like Tennessee Eastman Benchmark process, Powder Detergent Production Process and Thermal Oxidizer have been untouched. Grbić et al. (2013) developed a soft sensor for all the complex processes mentioned above using an adaptive mixture of Gaussian process regression (GPR). When results were analyzed, it was noticed that, when it comes to the nonlinear multimode processes, the adaptive mixture of GPR based soft sensors are better sensors than partial least square regression based soft sensors.
Novak et al. (2013) developed a soft sensor for the estimation of cold filter plugging point (CFPP) in a crude distillation unit. They developed the soft sensors using a genetic algorithm (GE) and adaptive neuro-fuzzy inference system (ANFIS) techniques. The input data was firstly preprocessed for the removal of outliers, missing data, and filtering of the data. After that, a soft sensor was developed for the online estimation of CFPP. It was observed that above techniques were significantly efficient for the nonlinear systems having complex processing.
The main difficulties observed in a soft sensor are that its performance deteriorates with time, and its accuracy also get reduced when sudden changes and non-linearities are observed in a system. Kim et al. (2013) developed a soft sensor for the solution of the above problems. They proposed a soft sensor based on the locally weighted partial least square technique to predict the aroma composition in cracked gasoline fractionator. The LWNPLS was used to handle the non-linearities by determining the strength of the nonlinearity in every input-output data and weigh it accordingly. The results obtained were very impressive giving more strength to the model against non-linearities.
Kaneko and Funatsu (2013) presented the soft sensors based on partial least square (PLS) technique with various adaptive models. The models that were studied with PLS were moving window (MW), just-in-time (JIT) and time difference (TD) adaptive models. They studied degradation of the soft sensors with the changes in the input variables ,x, and output variables ,y, of a process model. They categorized degradation as the change in x variables only, change in y variables only and change in the slope between x and y variables. They have also studied the response at instant or gradual changes in variables. When applied to an industrial problem, the results described that all the sensors worked fine at particular degradation situation. The TD model was best suited for instant changes in x values or y values while MW worked well during gradual changes

in the slope of x and y values. If the slope of x and y values are changing instantly with the change in x values and when the amount of fresh data is enough for the system than JIT model suites best else MW outperforms JIT model and TD model.

Earlier Wang et al. (2013) applied the non-negative garrote (NNG) variable selection technique to refine the inputs variables for the partial least square (PLS) based soft sensor. Sun et al. (2014) extended the use of NNG and used it to shrink the weights for a artificial neural network (ANN) based soft sensor. They used NNG with ANN based soft sensor to estimate the O_2 composition for the air separation process. They compared the results of NNG-ANN with the other methods such as sequential backwards multi-layer perceptron (SBS-MLP), ANN, and iterative ANN. Results showed that ANN individually was not better than other methods, but when NNG was integrated with the ANN technique, it produced better results than other methods and concluded that better variables selection technique can make a soft sensor more robust and more accurate.

As earlier neural networks were taken as black box model only, Mohamed Ramli et al. (2014) came with the idea of Equation-based Neural Network (NN). Equation-based NN is more reliable, robust, fast and concrete technique when applied to develop a soft sensor. They followed the method to develop a soft sensor and used it for the prediction of the n-butane composition in the debutanizer column. For the comparative studies, above method was compared with the partial least square (PLS), regression analysis (RA) and the artificial neural network (ANN) methods, and it was found that equation based NN was the best among other methods.

Developing a soft sensor using principal component analysis (PCA), partial least square (PLS), artificial neural network (ANN), or support vector machine (SVM) always carry some drawbacks in the process. As PCA and PLS are linear methods, they don't work for non-linear systems. Another problem carries that they both require a lot of data to get a reasonable accuracy for the sensor. However, ANN solved the problem of non-linearity, but it came with another drawback of uncontrollable speed convergence, and getting stuck at local minima. The problem of local minima was solved using SVM. But SVM drawback was its computational complexity which grows exponentially with the number of training samples, and both ANN and SVM were not allowing the space for latent variables which leads to the weak capability of interpretation. Shang et al. (2014) applied a technique which solved approximately all the above problems that were encountered by the other methods. They developed a soft sensor based on deep neural network (DNN) which is an application of deep learning studies. In DNN Firstly, the inputs of the network were sorted hierarchically by making a networked layer called deep belief network (DBN). The DBN was created using stacks of Restricted Boltzmann Machines (RBM). An RBM converts the input variables into latent vectors so that only useful information shall pass through the network. The last layer of the RBM transfers the information to last hidden layer of network. From that last layer, the output is generated by minimizing the gradient error. Benefits of using DNN technique was that it converts the inputs into latent vectors which lead to the rejection of less useful data, it is suitable for non-linear systems, its computation complexity increases linearly not exponentially as SVR encounters and it does not get stuck into local minima. They applied this technique to develop a soft sensor for the estimation of ASTM 95% cut point of heavy diesel in a crude distillation column. The results revealed that DNN gave better results than ANN, PLS, SVM and neural network PLS (NNPLS).

Yuan et al. (2014) concentrated on adaptive nature of a soft sensor to handle future changes in the process. They developed a soft sensor based on just-in-time learning (JITL) based Locally Weighted Kernel Principal Component Regression (LWKPCR) and applied it to two industrial processes. The JITL role was to adapt the changes of the processes with time and locally weigh the kernels which convert linear PCR to a non-linear technique. They compared JITL-based LWKPCR with JITWL-based PCR and KPCR. It was observed that JITL-based LWKPCR had the smallest root mean square error, proving to be the best among rest two techniques.

Liu (2014) presented a soft sensor for the estimation of O_2 composition in the air separation process. He introduced a technique of sparse partial least square with variable importance in projection (SPLS-VIP) to develop a soft sensor. He concentrated on the selection of quality variables and dynamic nature of the model when the input is having different time delays. The less useful information is rejected using both SPLS and VIP technique, and the sparse nature of PLS was used to make the approach dynamic in nature. The results revealed that SPLS-VIP technique is better and more accurate approach than PLS, SPLS and PLS-VIP when applied on air separation process.

Kaneko et al. (2014) explored adaptive models for the partial least square (PLS) based soft sensor. They opted moving window (MW), just-in-time (JIT) and time difference

(TD) adaptive models. In MW reconstruction of the model takes place with the most recent data while, In JIT modeling the model reconstruction is based on the similarity of the data with output data. The TD concentrates on changes which are gradually taking place in the process with respect to time. The PLS based sensor with JIT, TD and MW were studied on depropaniser distillation column for the estimation of bottom product composition. It was observed that TD model worked best on steady state models, while MW and JIT based models were preferred for nonsteady conditions.

Ge (2014) extended the old fashioned principal component regression (PCR) based soft sensor and developed smart soft sensor based on PCR. Smart soft sensor labels the data according to the significance and information contained in the data which might be useful for the output prediction. The objective function of the sensor was to minimize the number of samples which were having labels on it. This sensor was applied to predict the product composition for the debutanizer column, and it was noticed that smart soft sensor gave considerable accuracy when compared with the real data.

The support vector regression (SVR) has been proved to be an accurate technique with few drawbacks. Behnasr and Jazayeri-Rad (2015) extended SVR to iteratively weighted least square SVR (IWLSSVR) technique which is more robust than conventional SVR. They collaborated nonlinear autoregressive with exogenous inputs with the least square SVR (LSSVR) method for the estimation of product composition in a debutanizer column. Results of the above developed soft sensor were compared with Adaptive Neuro-Fuzzy Inference System (ANFIS) technique based soft sensor, and it was observed that IWLSSVR outperformed ANFIS in the presence of outliers and noises in the input data.

Zhu et al. (2015) presented a soft sensor technique for the systems that have outliers in their variables and also have multiple operating conditions. They developed a mixture of robust supervised probabilistic principal component analysis (MRSPPCA) model and applied it to two case studies. In above method, Outliers were handled by adjusting the heavy tails using multivariate student t-distribution. After removing the outliers, iterative expectation-maximization algorithm was used to estimate the parameters for the models. When MRSPPCA and PPCA based soft sensor were compared, it was observed that MRSPPCA gave more accurate, stable and robust results than PPCA based soft sensor.

Shao et al. (2015) developed a soft sensor to handle the time varying and nonlinear characteristics of the process. They developed a soft sensor based on local partial least squares models with adaptive process state partition (LPLS-APSP) to estimate product composition in a CSTR reactor and debutanizer column. In this technique, the global model was converted into local models whose window of partition was decided according to the time intervals and then all the local models were solved using PLS technique. The results showed that LPLS-APSP gave accurate results with very less prediction error.

Shang et al. (2015) developed a soft sensor based on the dynamic partial least square (DPLS) technique to predict the ASTM 95 % cut point of heavy oil for a crude distillation column. They wanted to study the dynamic and nonlinear features of the process, so they used lagged inputs to make it dynamic. The problem of over-fitting was deteriorating the performance of a soft sensor and for that, they proposed a temporal smoothness regularization technique. The smoothness regularization assumes that the historical data is a real data and any later abrupt change will be considered as a disturbance. This method made the soft sensor more generalized in nature. Along with crude distillation column, the soft sensor was also applied to Tennessee Eastman process study and it was observed that soft sensor based on above technique worked efficiently for predicting the output.

Earlier Kaneko et al. (2014) discussed three adaptive soft sensor models. Kaneko and Funatsu (2015) extended the study of adaptive models by joining them to increase the adaptive accuracy of the sensor. They developed a soft sensor based on partial least square (PLS) with Time difference moving window (TDMW) and time difference just-in-time (TDJIT) model. These combinations were made to deal with non-linearity of the model. When the above techniques were applied to an industrial problem of estimating phenol composition in the distillate of the phenol production column, results proved that both techniques worked well. Whereas, TDJIT showed some instability when both input and output variables vary simultaneously.

Gholami and Shahbazian (2015) developed a soft sensor to predict the H_2S composition in sweetening stripper column. They proposed fuzzy C-means clustering with the recursive finite newton algorithm to train the support vector regression (FCM RFN SVR) technique for the development of soft sensor. The role of FCM was to cluster the unsupervised data and make partitions, and RFN SVR was used to predict the output and for solving all the local models. The results showed that above technique performed substantially well for estimating the composition and gave good generalization capability to the model.

Jalee and Aparna (2016) developed a soft sensor for the estimation of top and bottom product composition of a distillation column. They developed the soft sensor based on NARX based ANFIS technique for the estimation purpose. They estimated the composition using secondary variables such as tray temperatures, reflux rate and reboiler duty. The data was collected from the ASPEN HYSYS software for the training and testing of the network. The results of ANFIS based soft sensor estimations were compared with neural network based sensor results and it was observed that the ANFIS gave more accurate results with small MSE and high R^2 value than neural network based soft sensor predictions.

Yan et al. (2017) developed a soft sensor for the estimation of butane composition in the distillate stream using tray temperatures, reboiler duty, reflux flow, and liquid flow rate. They carried out the study of soft sensor using weighted linear dynamic system technique (WLDS). The WLDS included the non linearity and dynamic behavior of the plant in better way than latent variable models. The WLDS proposed two types of weights for local linearization of the nonlinear state models and approximations of state emission. The WLDS was linked with the expectation maximization (EM) technique for the estimation of parameters of debutanizer column. The results showed that the proposed method gave better results with small root mean square error (RMSE) when WLDS estimations were compared with probabilistic principal component analysis(PPCA) and weighted PPCA (WPPCA) results.

After Shang et al. (2014), Yuan et al. (2018) used the deep learning (DL) based neural networks for the soft sensing purpose. They developed a soft sensor based on DL networks which includes the denoising autoencoders with the neural network (DAE-NN) technique to estimate O_2 composition in a flue gas in ultrasuperficial plant. The benefit of deep learning technology is its property of narrowing the information enclosed in input variables till last layer. They observed that the DL based soft sensor gave better generalized results with small error when compared with real values of O_2 in flue gas.

2.2 Soft Sensors for Reactive Distillation Columns

The work have also been done on developing a soft sensor for reactive distillation column (RDC). As composition decides the conversion of reaction and efficiency of the column, the on-line monitoring of composition plays a crucial role in RDC. Many soft sensors were developed for estimating the product composition in RDC. Some of the literature survey on soft sensor for RDC has been given below

Olanrewaju and Al-Arfaj (2006) proposed the first soft sensor for on-line monitoring of reactive distillation column (RDC). As RDC is a complete non-linear process, they preferred a non-linear technique for online estimation. They developed extended Kalman filter (EKF) based soft sensor for estimating the distillate and bottom product composition of an RDC. They concentrated on making soft sensor more robust. The results proved that above soft sensor worked correctly with noisy measurements, and uncertain initial conditions.

Venkateswarlu and Jeevan Kumar (2006) studied extended Kalman filter technique to develop a soft sensor for a reactive distillation column. Firstly they proposed principal component analysis (PCA) technique for the input variable selection and then EKF for soft sensing. In RDC the esterification reaction was studied for the production of ethyl acetate. The results showed that combination of EKF and PCA proved to be robust technique for the online monitoring of RDC. The soft sensor also provided the advantage of speedy and reliable convergence in the presence of noisy data.

As Olanrewaju and Al-Arfaj (2006) and Venkateswarlu and Jeevan Kumar (2006), Sumana and Venkateswarlu (2009) also worked on extended Kalman filter (EKF) but they opted another variable selection technique. They used empirical observability gramian for the input variable selection. They developed an observability covariance matrix, which reflects the influence of the input variables on the model output and proposed a soft sensor to estimate the composition of top, bottom and intermediate composition in RDC. When the result of the above proposed soft sensor were compared with real data, it was observed that results were satisfactory.

Bahar and Özgen (2010) worked on estimation and control of composition for the reactive distillation column. Firstly, they developed an ANN based soft sensor for the estimation of product composition in RDC and then developed an inferential controller to control the composition in the column. They used Elman based network having two hidden layers. When results were obtained, it was observed that combination of inferential estimator and controller worked fine for the reactive distillation column.

Developing a soft sensor based on extended kalman filter was the standard technique, but it was having a drawback that its performance depends on thermodynamical modeling of the process. To solve this problem, Khazraee and Jahanmiri (2010) proposed a soft sensor which was the combination of artificial neural network and fuzzy logic techniques i.e. Adaptive Neural Fuzzy Inference System (ANFIS). They developed ANFIS based soft sensor to predict the distillate composition in a RDC and it was observed that ANFIS based soft sensor gave better results without any need for a thermodynamic model. It was also observed that ANFIS based soft sensors were easy to initialize and tune.

Khazraee et al. (2010) extended the study of ANFIS to ANFIS with differential evolution (DE) optimization technique. The optimization method was used to optimize the functioning policy of the RDC. An ethylacetate esterification reaction was studied in the batch reactive distillation column. When soft sensor was developed for the estimation of the product composition, it was observed that above technique reduced the CPU usage to $1/18000$ of its original usage and more robustness was also seen in the model.

Vijaya Raghavan et al. (2011) developed a dynamic soft sensor based on recurrent neural network (RNN) technique for the reactive distillation column. In RNN technique, estimation is not only dependent on current input readings but also on the last time reading. It was observed that they do not recurrent the previous reading, leading to the use of dynamic neural network instead of RNN. They proposed time-delayed neural network (TDN) based RNN to estimate the distillate composition of the RDC. They integrated RNN based soft sensor with a PI controller and studied both open loop and closed loop responses for the model. When RNN based soft sensor technique was compared with EKF and FNN based soft sensor, RNN clearly outperformed other two soft sensors. It was also observed that RNN based soft sensor does not require any prior knowledge of the disturbances and it is inconsiderate to the model parameters.

Sakhre et al. (2016) developed a control structure for an RDC having a soft sensor as a measuring element in the closed loop. They proposed a feed forward neural network trained back propagation (FBPNN) technique based soft sensor for the estimation of product purity of an RDC. The network was trained using steepest descent optimization technique for the better minimization of MSE during the training of the network. They used the soft sensor as a measuring element in a single loop and a cascade loop control of RDC. In the case of cascade control loop, outer loop was manipulating last tray temperature and inner loop was manipulating reboiler duty of the column. They observed that the cascade control results were having less sluggish response than single loop based control response and concluding its superiority over single loop control strategy.

Jana and Banerjee (2017) designed a control system with neuro estimator based inferential extended generic model controller (IEGMC) linked with ANN based soft sensor to control the ethylene glycol composition in an RDC. The estimations were carried out using the most sensitive tray temperatures as the input variables to estimate the product composition in an RDC. It was observed that soft sensor estimations were comparably good with the real values of composition. The IEGMC also gave better set point tracking and disturbance rejection results when compared to inferential proportional integral (IPI) controller.

2.3 Study of Inverse Response

The inverse response occurs when two opposing processes occurs at a same time. The response during this condition changes its track from its initial direction and ends up in the opposite direction. The inverse response causes sluggish behavior and instability in the closed loop. The inverse response problem has been experienced and addressed in various chemical units. Some of the literature survey has been mentioned as follows.

Iinoya and Altpeter (1962) discussed the problem of an inverse response for the first time. They gave the general introduction about an inverse response occurring due to step change in manipulating variable, its causes and its solution. They developed a compensator which removes the positive zeroes from a 2^{nd} order process containing two 1^{st} order opposing processes. They also mentioned other transfer functions which can experience the problem of inverse response due to the presence of positive zeroes in the transfer functions.

Rovaglio et al. (1996) handled an inverse response in municipal incineration plant. In the municipal incineration plant, when a positive step change is given to the waste flow rate, the steam flow rate shows inverse response leading to the high oscillations in the closed loop. The problem was handled by developing a compensator for a 2^{nd} order transfer function model of municipal incineration plant. The closed loop having PI controller coupled with a compensator gave more stable and less oscillatory results as compared to a PI controlled closed loop.

Scali and Rachid (1998) dealt with the problem of inverse response in a 2^{nd} order transfer function model using an internal model control (IMC) technique. The IMC holds the benefit of less complexity in design as compared to inverse response compensator. The IMC also holds the benefit of getting easily tune using a single parameter. They observed that the IMC technique gave better and stable results for set point tracking and load changes. They also concluded that the IMC controlled closed loop is robust to the model uncertainties.

The inverse response in a CSTR was observed by Camacho et al. (1999). They handled the problem of inverse response using a sliding mode controller. The controller was developed from a first order plus dead time model of the process. They observed that the controller is well suited for high order non linear and complex models. The results of the controller showed high stability in the closed loop response during the presence of various disturbances, modeling errors and noises in the process.

Luyben (2000) also dealt with the inverse response in the CSTR but they expanded the problem by including the problem of dead time in the system. They converted the CSTR process into a 2^{nd} order transfer function model and then solved the problem of inverse response and dead time using a PI controller. They tuned the PI controller by taking the parameters of the controller as a function of positive zeroes and dead time. They compared the proposed tuning technique based PI controller with the Ziegler Nicholas (ZN) tuning based PI controller. The comparative study showed that the proposed technique gave better results with less overshoot and high stability than ZN method.

The compensator designed by Iinoya and Altpeter (1962) is based on the working of Smith predictor which is a dead time compensation technique. Zhang et al. (2000) extended the smith predictor for the inverse response problem for two simultaneously working 1^{st} order transfer function models. They developed the smith predictor using H_∞ theory and compared the results with internal model control based technique. The results obtained were more robust than conventional compensator results. They concluded that the proposed compensator can also be applied to higher order unstable processes.

Chien et al. (2003) addressed the problem of inverse response in the a 2^{nd} order plus dead time transfer function models. They used a simple PI/PID controller tuning technique which is based on the direct synthesis method. They observed that the proposed controller gave better results with small overshoots when compared with other PID controllers. They checked the controller stability at various model mismatch condition and found the controller to be robust to the model uncertainties. They extended the study of the proposed controller for controlling a highly nonlinear distillation column. The closed loop results of controlling a column was satisfactory for all the set point and load change conditions.

Luyben (2003) studied the inverse response problem in the boiler drum. He studied the identification and controller tuning technique for PID controller and concentrated on minimizing the integral action to reduce the complexity in the process. They observed that the proposed method is easy to use and gives reliable closed loop response. The also mentioned that the proposed technique holds a drawback of not getting reliable results at high noise levels.

Sree and Chidambaram (2003) discussed two PID tuning techniques for the problem of inverse response in the CSTR with and without delay. The first method contained two tuning parameters and second contained a single tuning parameter. They compared the closed loop results of both the proposed techniques and technique opted in Luyben (2000). They observed that the first proposed technique provided better stable results at both the servo and regulatory response plus in the presence of model uncertainties in model gain, time constants and zeroes location.

The processes with the problem of inverse response is difficult to handle than processes with dead time. Skogestad (2004) proposed the solution of inverse response by transforming the inverse response time constant into a time delay. He studied a model reduction technique which reduces the process order by defining an effective dead time and then tuning a PI/PID controller using a single tuning parameter. The proposed technique gave smaller integral of the square error (ISE), smoother response and robust controller settings.

Chen et al. (2005) also opted the conversion of inverse response characteristics into dead time. They transformed the inverse response right half point zeroes into dead time using Pade approximations. They considered a generalized 2^{nd} order transfer function with the inverse nature for the study. They used a single parameter based PID controller which was based on the conventional unity feedback control. The results showed that the problem of inverse response was eliminated from the process using proposed technique and the control results are robustly stable in nature.

Gu et al. (2006) discussed the problem of an inverse response and dead time in a process such as boiler drum. They eliminated the problems using an auto tuned PI/PID controller which is a single parameter based controller designed using H_∞ theory and IMC control theory. The applied the technique to a 2^{nd} order transfer function model having dead time in the process. They observed that the proposed controller gave stable, fast and less oscillatory results than ZN tuned controller and Luyben (2000) based controller. The single parameter PI/PID controller is also robust to the model uncertainties.

Jeng and Lin (2011) dealt with the problem of inverse response and dead time using a PID control technique which is based on the working of a Smith predictor. The considered a 2^{nd} order transfer function model with a dead time in the process and developed a control scheme with a classic PID controller designed using Maclaurin series approach. The PID is tuned using a single parameter by balancing the robust stability and better performance of the closed loop . The designed PID gave better robust results with less overshoot and small oscillations when the response is compared with ZN, Chien et al. (2003), and Chen et al. (2005) based PID controllers response.

In literature the work was majorly done on PID controller of 1 degree of freedom (DoF) but Alfaro and Vilanova (2013) extended it to a PID controller with 2 DoF. They handled the problem of inverse response using 2 DoF PID controller having five parameters. They considered a second order plus right half plane zero (SOPRHPZ) transfer function model and controlled it using PID controller which is tuned using model reference technique. The controller gave robust, stable and better results with small undershoots and smoother response when compared to the PID tuned using Scali and Rachid (1998) technique.

Kaya (2016) mentioned the problem of inverse response to be more serious and complicated than dead time problem. They started by eliminating the factor of inverse response in the process using factorization of transfer function. Then they developed a PI-PD controller which is tuned using algebraic tuning approach. They proposed that, a

PID controller cannot handle the troublesome nature of the inverse response adequately. When the controller is applied to a 2^{nd} order transfer function model, the results showed that the PI-PD controller gave more satisfying results when the proposed methodogy is compared with Chien et al. (2003) technique.

2.4 Model Predictive Control (MPC)

The MPC is an advance control technique which control the process by optimizing the plant input according to the future model predictions of the process. The MPC primary components are its model and optimizer. The literature shows various types of models and optimizers used in the MPC to control the variety of chemical units such as distillation column, reactor etc. Some of the literature survey on MPC is as follows.

Giwa and Karacan (2012) applied the MPC technique to control the top, middle and bottom tray temperatures of packed reactive distillation column. They manipulated the reflux ratio, feed ratio and reboiler duty of the column for the control of tray temperatures. They developed the decoupling based MPC with neural network based model and transfer function based model inside MPC. They applied the developed controllers on the experimental study and found out that neural network based decoupling MPC are better performer than transfer function based decoupling MPC. The results were verified by the small integral of square of error (ISE) during neural network based MPC.

Rewagad and Kiss (2012) developed an MPC for the control of a product composition in a divided wall column (DWC). They used a linear state space model inside the MPC which was obtained by the linearization of the non linear model of DWC. They compared the control structures of DWC having only PID controller, only MPC controller and combined PID and MPC controllers. They observed that the MPC provided better results than PID but similar results with the MPC and PID combined control scheme. They added white noise to the model to check the robustness of the controller and proved that MPC is robust technique with better stability.

Heidarinejad et al. (2012) designed an MPC for a non-isothermal CSTR by focusing on the economics of the process. They designed an economic MPC which is based on Lyapunov based control techniques. Instead of going with the conventional cost function they transformed the cost function that works in two steps. The first step involves optimization of cost function around the stable closed loop. In second step the MPC takes the process into an adequate steady state condition by keeping the system into confined constraints. They observed that MPC provides stable and robust results even in the presence of maximum time delay.

The inside model of the MPC plays a crucial role in the control of a nonlinear process has been validated by Sharma and Singh (2012). They proposed a neural network predictive controller to control the tert-amyl methyl ether composition in an RDC. They used the inside model of MPC to be an artificial neural network model of RDC which encloses all the nonlinear properties of the column. They observed that the neural network based MPC outperformed transfer function based MPC with small integral error values.

Martin et al. (2013) pointed out that a linear model of the process changes during the varying operating points. They proposed a multi model MPC for controlling two tray temperatures of pilot distillation column by manipulation of the reboiler duty of the column. The state space linear model was considered for the study. They concentrated on the problem of dead time and multi-poles problem in the system. The results were robust in nature during the use of multi-model MPC for a distillation column.

Lopez-Negrete et al. (2013) presented a nonlinear MPC for the control of a binary distillation column and steam generation unit in a power plant. They developed an advanced step nonlinear MPC (asNMPC) which optimizes the cost function in the background of the process and applies the sensitivity based updates at the real time. The asNMPC is removing a major problem of computational burden and complexity by the order of two to three folds. The results showed that the closed loop response of asNMPC is more robust and stable than conventional NMPC controlled process.

Major work has been done on the MPC by studying theoratical models of the plant limiting its use for theoratical studies rather than experimental studies. Huyck et al. (2014) developed an MPC and connected it to a pilot distillation column using appropriate hardware. They developed an online MPC and connected the MPC with distillation column using Programmable automation controller (PAC) and Programmable logic controller (PLC). They considered the linear state space model inside the MPC. They studied two types of quadratic programming (QP) for the optimization purpose that is Hildreth algorithm and the qpOASES algorithm. They observed that PAC works on both the optimization procedures but PLC works only on Hildreth algorithm.

Vu (2015) studied the MPC for the removal of inverse response in the process. He considered a 4^{th} order discrete transfer function with the inverse response for the study. Since the MPC with finite horizon can only remove the inverse response partially he considered an MPC with infinite control horizon to remove the problem of inverse response completely from the system. He observed that the controller with 2 DoF completely rejects the inverse response from the system while 1 DoF was unable to remove it completely.

Biegler et al. (2015) extended the study of asMPC as earlied done by Lopez-Negrete et al. (2013). They extended the asMPC to a dynamic real time optimization (D-RTO) for focusing on the economic structure of the process. They considered this study for the control of product composition in two large scale distillation columns. They used the technique of nonlinear programming for the optimization purpose which decreased the computation load of CPU by 2-3 times lesser than conventional processes. It was observed that the results were more stable for handling the set changes in the system.

Yamashita et al. (2016) solved the problem of tuning MPC for a Crude Distillation Unit (CDU). A multi objective cost function has been developed for controlling the CDU using an MPC. The outputs of the CDU are controlled by assigning them a particular zone instead of a single set point. The MPC works by penalizing the input moves and output error if they deviate from their target values. The proposed method was compared with the other multi objective technique based on posteriori solution which is 22 time more computationally expensive than proposed technique.

Oravec et al. (2016) applied the study of alternative robust MPC to control a lab scale heat exchanger. They transformed the optimization problem and their constraints into a linear matrix inequality which was solved using a semi definite programming (SDP). This leads to the better participation of influential uncertain parameters into the control work. They designed a controller and implemented the controller using a MUP toolbox of MATLAB® software. They found out that the proposed MPC gave more better, robust and stable control results than the earlier advanced MPC methods.

Serrezuela and Chavarro (2016) developed a multi-variable MPC which was used to control a distillation column and an evaporator. They compared the proposed MPC results with multivariable decoupling control, multiloop control, both having PI structure, a decoupling control based on Inverse Nyquist Array, and an IMC. They studied the effect of dead time and the capability of the controller of rejecting the disturbances. They observed that the proposed MPC proved to be most superior than other control methods and removed the problem of sluggishness in the system.

Mahindrakar and Hahn (2016) studied the control of a reactive distillation column having benzene hydrogenation reaction in the column. It was studied from the literature that controlling an RDC having benzene hydrogenation reaction causes sluggish response during a step change in feed flow rate while using a decoupled PI controller. They proposed a feedforward feedback controller which gave better results but with the problem of costly composition analyzer for the control. Then they proposed a SISO and MIMO MPC for the control of RDC having transfer function models inside the MPC. They observed that SISO MPC provided better results than MIMO MPC and removed the sluggish response of the closed loop.

He et al. (2016) controlled a CO_2 capture plants using an MPC technique. Firstly they proposed a linear model MPC which needs to be tuned at high frequencies to solve the problem of unwanted oscillation in the closed loop. Then they proposed an integrated scheduling and control framework for minimizing the cost of the operation with maximum CO_2 removal from the plant. They observed that the proposed methodology gave more economic results than conventional scheduling and control results.

Oh and Lee (2016) studied an iterative learning MPC (ILMPC) for controlling a nonlinear batch reactor. From the literature it was observed that a iterative learning controller (ILC) hold a drawback of its inability to reject the real time disturbances. The problem was solved by combining both ILC and MPC techniques for controlling the reactor at real time disturbances. It was observed from the results that the proposed ILMPC technique can easily reject the dynamic real time disturbances and also holds stability during model uncertainties.

2.5 Conclusions

In this chapter, the literature survey on development of soft sensors, the problem of inverse response and development of model predictive controller was studied. The study involved the different types of soft sensors for the estimation of quality variables for various types of chemical units especially for distillation columns and reactive distillation columns. The literature included problem of inverse response for various chemical

units such as distillation columns, CSTR, and municipal incinerators and solved using various compensators and controller tuning techniques. In the case of MPC, the literature showed variety of work done on the different types of internal models and optimizer involved in the MPC for the control of quality variables in various chemical units.
The literature mainly includes the distillation columns in which either tray temperatures or composition is controlled directly without using any soft sensor. The soft sensors which have been used for composition control of distillation columns are not dynamic when they were developed using neural network techniques. Many reaction systems used in RDC need to be explored much for soft sensing purpose such as butyl acetate esterification reaction system. In the case of inverse response due to load variable, the solution of the problem has not been discussed yet. Although the problem of inverse response due to manipulating variable has been solved for 2^{nd} order or reduced 2^{nd} order transfer function models but not for higher order models. Literature generally discusses the control system including a soft sensor with a PID controller, however, coupling the soft sensor with the advanced process controller needs to be explored more.

Chapter 3

Mathematical Modeling of Reactive Distillation Column

The reactor followed by a distillation column for the product separation is a common practice in the chemical industries. This practice involves high inventory, energy and manpower which leads to high financial and technical burden on the industry. The alternative of this was to combine reactor and distillation column into single entity, leading to the phenomenon of reactive distillation (RD). The basic concept of RD is the integration of two processes, reaction, and separation, in a single still known as reactive distillation column (RDC). In spite of having two sections, rectifying and stripping, like in a conventional distillation, RDC includes third section named reactive section. This reactive section is enclosed with a packing embedded with a catalyst for the reaction purpose. One of the prime motive of choosing an RDC is to remove the products from the column as soon as they are being produced. This factor helps reversible reactions to restrict the backward reactions to take place. It leads to better selectivity, better conversion, better heat utilization and avoidance of azeotropes in the column.

In reversible reactions, esterification reactions are intensively studied in the literature. This study will involve esterification reaction in a reactive distillation column (Luyben and YU, 2008) for the production of n-Butyl acetate. Esterification reaction of n-Butyl acetate is shown as.

$$\underset{\text{Acetic Acid}}{\mathrm{C_2H_4O_2}} + \underset{n-\text{Butanol}}{\mathrm{C_4H_9OH}} \xrightleftharpoons{amberlyst15} \underset{n-\text{Butyl Acetate}}{\mathrm{C_6H_{12}O_2}} + \underset{\text{Water}}{\mathrm{H_2O}} \tag{3.1}$$

In the above reaction, an Acetic acid reacts with n-Butanol in the presence of catalyst Amberlyst 15 for the synthesis of n-Butyl acetate and water. The reaction is only taking place in the reactive section of the RDC which is embedded with catalyst packing as shown as in Fig. 3.1. The reaction is accompanied by separation process which takes place in all the three sections but predominantly in rectifying and stripping section of the column. In the column, stream 1 and 2 are the feed streams for the two reactants.

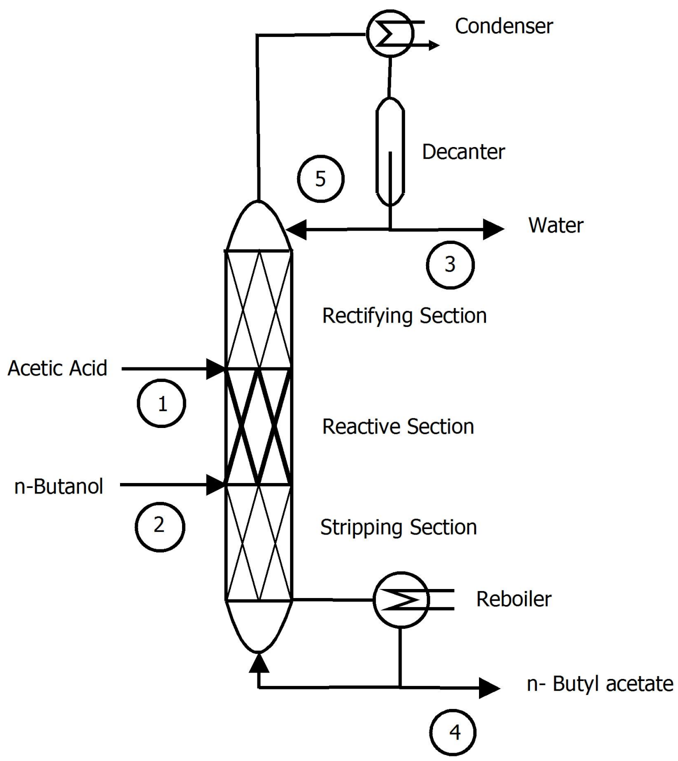


Figure 3.1: Reactive Distillation Column

Two different reactant streams are provided so that the temperature gradient among the trays leads the reactants to meet in the middle of the column for the reaction. As Acetic acid is heavier than n-Butanol, it is passed on the upper tray and butanol on the lower tray. After reaction and separation, products can be collected from stream 3 & 4. Water being lighter than n-Butyl acetate, is obtained as a top product while n-Butyl acetate as a bottoms product. Venimadhavan et al. (1999) mentioned that there is a need for a phase separator (Decanter) to separate the heterogeneous mixture of liquids comprising two phases in a distillate product stream. One phase is an aqueous phase with 100 % water and the second phase is an organic phase containing approximately 83 mole % of butyl acetate and 17 mole % of water. The condenser stream is sent to decanter unit where the separation of liquid phases takes place. Among both the decanter streams, the organic one is refluxed back to the RDC and aqueous stream is taken as the distillate product. The prime product of the reaction, n-Butyl Acetate, is separated from the column as a bottoms product.

For the modeling purpose, reaction kinetic model needs to be considered for the given reaction. In literature, mainly, pseudohomogeneous model has been considered as a reaction kinetics model for the butyl acetate esetrification reaction. The reaction kinetic model taken from Luyben and YU (2008) is as follows.

$$r = m_{cat}(k_F a_{HAc} a_{Buol} - k_B a_{BuAc} a_{H_2O}) \tag{3.2}$$

$$k_F = 3.3856 \times 10^6 \exp \frac{-70660}{RT} \tag{3.3}$$

$$k_B = 1.0135 \times 10^6 \exp \frac{-74241.7}{RT} \tag{3.4}$$

where the reaction rate, *r*, is in *kmol* s^{-1}, m_{cat} is mass of catalyst taken in *kg*, k_F and k_B are forward and backward rate constants considered in *kmol* $kg_{cat}^{-1} s^{-1}$, *a* is the activity of the respective component, *R* is gas constant taken in $kJ\ K^{-1} kmol^{-1}$ and temperature *T* is in *K*.

3.1 Modeling of RDC

The model of any process is very crucial for the planning and controlling the plant. In the case of advanced process control techniques, they majorly work on the basis of process model of the plant, thus the model accuracy plays an important role in it.
In this chapter, a steady state and a dynamic model of the reactive distillation column has been developed in MATLAB® and SIMULINK® software. The model involved all the material, component and energy balance equation with various other distillation concepts and calculations. The model is solved using ODE15s solver of MATLAB® which is basically an ordinary differential solver of stiff differential equations. The model has been validated using other CHEMCAD® software based simulated model and with the experimental study of Steinigeweg (2002). The study of open loop response of the model with further complications of inverse response are also included in this chapter.

3.1.1 Assumptions

The model of any plant starts with some assumptions, which we presumed that are true for the model. The assumption taken for the presented model can be enlisted as.

1. The reaction is taking place only in reactive section as the catalyst is only embedded in reactive section.

2. Since most of the catalyst is in the contact of liquid, the reaction is assumed to be occurring only in liquid phase.

3. As the column works at atmospheric pressure, the vapor holdup is assumed to be negligible as compared to liquid holdup. As in this case the vapor density is very low as compared to liquid density.

4. As per the literature survey the UNIQUAC is considered for a thermodynamic modeling.

5. The Pressure drop along every tray is assumed to be negligible.

6. The levels in reflux drum and bottom of the column have been considered to be perfectly controlled.

7. The fluid properties have been assumed to be constant.

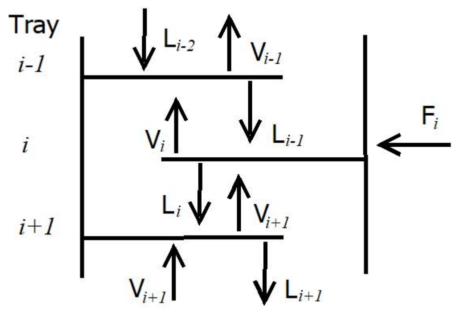


Figure 3.2: Material balance on i^{th} tray

3.1.2 Dynamic Model Equations

In the modeling of RDC, NT number of trays are taken for the system with NC number of components. The stages are numbered from top to bottom, where, condenser is taken as 1^{st} and reboiler is NT^{th} number of tray. The material balances on each tray has been done according to Fig. 3.2, while for the component and energy balance, respective composition and enthalpy term is multiplied with molar flow rates. All the balance equations for the column are

Material balance on i^{th} tray

$$\frac{dM_i}{dt} = L_{i-1} + V_{i+1} - L_i - V_i + F_i \tag{3.5}$$

where, L_i, V_i and F_i are liquid, vapor and feed flow rates on i^{th} tray in mol/h. M_i is a molar holdup on i^{th} tray.

Component material balance on i^{th} tray and j^{th} element

$$\frac{dM_i x_{i,j}}{dt} = L_{i-1}x_{i-1,j} + V_{i+1}y_{i+1,j} - L_i x_{i,j} - V_i y_{i,j} + F_i x_{fi,j} + \Delta R_i \tag{3.6}$$

where, $x_{i,j}$ and $y_{i,j}$ is a liquid and vapor mole fraction of j^{th} component on i^{th} tray. ΔR_i is a rate of reaction on i^{th} tray in mol/h and is estimated using Eq. 3.2.

Enthalpy Balance on i^{th} tray

$$\frac{dM_i h_i}{dt} = L_{i-1}h_{i-1} + V_{i+1}H_{i+1} - L_i h_i - V_i H_i + F_i h_{fi} \tag{3.7}$$

where, h_i and H_i are liquid and vapor enthalpy of mixture on i^{th} tray in J/mol. The heat of reaction is enthalpy of formation of products minus enthalpy of formation of reactants. In the energy balance equations of the trays, components enthalpies have already been included in the equation. Therefore, there is no need to include extra heat of reaction term into the equation.

Material Balance on Condenser (1^{st} Tray)

$$\frac{dM_1}{dt} = V_2 - R - D \tag{3.8}$$

where, R is reflux rate and D is Distillate Rate.

Component Material Balance on Condenser

$$\frac{dM_1 x_{1,j}}{dt} = V_2 y_{2,j} - (R + D)x_{1,j} \tag{3.9}$$

Enthalpy Balance on Condenser

$$\frac{dM_1 h_1}{dt} = V_2 H_2 - (R + D)h_1 - Q_C \tag{3.10}$$

where, Q_C is condenser duty in J/h.

Material Balance on Reboiler (NT^{th} tray)

$$\frac{dM_{NT}}{dt} = L_{NT-1} - V_{NT} - B \tag{3.11}$$

where, B is bottom mole flow rate.

Component Material Balance on Reboiler

$$\frac{dM_{NT} x_{NT,j}}{dt} = L_{NT-1}x_{NT-1,j} - V_N T y_{NT,j} - B x_{NT,j} \tag{3.12}$$

Enthalpy Balance on Reboiler

$$\frac{dM_{NT} h_{NT}}{dt} = L_{NT-1}h_{NT-1} - V_{NT+1}H_{NT+1} - B h_{NT} + Q_B \tag{3.13}$$

where, Q_B is reboiler duty in J/h. The distillate and bottoms flow rate can be estimated from material balance equation of condenser and reboiler.

$$D = V_2 - F_{NF_1} \tag{3.14}$$

$$B = L_{NT-1} - V_{NT} \tag{3.15}$$

where F_{NF_1} is an organic phase reflux stream sent by decanter into the column.

3.1.3 Parameters in the Model

After describing the balance equations on each tray, the parameter needs to be estimated which are involved in the model equations. These includes liquid and vapor flow rates, tray temperature and vapor compositions on each tray, liquid and vapor enthalpies.

3.1.3.1 Liquid and Vapor Rates

The liquid flow rate on each tray is estimated using residence time of the liquid holdup at each tray. It is assumed that the liquid holdup residence time on each tray is 5 seconds. The liquid flow rate in $mol\ h^{-1}$ on i^{th} tray can be estimated as.

$$L_i = \frac{\sum_{i=1}^{NT}\sum_{j=1}^{NC} m_{i,j} \times 3600}{5} \tag{3.16}$$

The vapor mole flow rate on each tray is estimated using an assumption of fast energy equation.

$$\frac{dh}{dt} = 0 \tag{3.17}$$

As total condenser is used in the column, vapor rate of the 1^{st} tray is equals to 0 and the vapor rate of the reboiler can be estimated from Eq. 3.13.

$$V_{NT} = \frac{L_{NT-1} \times (h_{NT-1} - h_{NT}) + Q_B}{H_{NT} - h_{NT}} \tag{3.18}$$

After estimating V_{NT}, the vapor rates for i_{th} tray can be estimated from Eq. 3.7 as.

$$V_i = \frac{V_{i+1} \times (H_{i+1} - h_i) + L_{i-1} \times (h_{i-1} - h_i) + F_i * (hf_i - h_i)}{H_i - h_i} \tag{3.19}$$

where F_i is feed stream for reactant feed stages and reflux stream for 2^{nd} stage with the feed tray enthalpy of hf_i.

Using Eq. 3.17 in Eq. 3.10, the condenser duty can be estimated as.

$$Q_C = V_2 \times (h_1 - H_2) \tag{3.20}$$

3.1.3.2 Tray Temperature and Vapor Composition

The temperature and vapor composition of all the trays can be estimated using the initial approximations of liquid composition and pressure on each tray. The concepts of modified Raoult's law, Antoine equation and BUBL T calculations are used for the calculations.

Initially, liquid composition and pressure on the tray is used to estimate the saturated tray temperature using Antoine Equation.

$$\ln(P_j^{sat}) = A_j - \frac{B_j}{T_i + C_j} \tag{3.21}$$

where, P_j^{sat} is in kPa and T_i is in K. The Antoine coefficients for the above equations

Table 3.1: Antoine Coefficients

	A	B	C
Acetic Acid	14.793	3405.57	216.81
Butanol	15.201	3137.02	178.72
Butyl Acetate	14.168	3151.09	203.87
Water	16.288	3816.44	227.02

are given in Table. 3.1. Using the values of P_j^{sat} and T_i the vapor composition is estimated using modified Raoult's law.

$$y_{i,j} P = x_{i,j} \gamma_{i,j} P_{i,j}^{sat} \tag{3.22}$$

where, $\gamma_{i,j}$ are the activity coefficients which are the measure of deviations of the com-

Table 3.2: UNIQUAC Area and Volume Parameters

Component	r	q
Acetic acid (1)	2.2024	2.072
n-Butanol (2)	3.4543	3.052
n-Butyl acetate (3)	4.8274	4.196
Water (4)	0.9200	1.400

pound from ideality. The activity coefficients are dependent on the temperature and composition of the compound. For the butyl acetate esterification reaction, UNIQUAC is used as a thermodynamic model for the calculation of the activity coefficients. The UNIQUAC model can be shown as.

$$\ln \gamma_i = \ln \gamma_i^C + \ln \gamma_i^R \tag{3.23}$$

$$\ln \gamma_i^C = \ln \phi_i + l_i + \frac{z}{2} q_i \ln v_i - \phi_i \sum_{j=1}^{nc} x_j l_j \tag{3.24}$$

$$\ln \gamma_i^R = q_i \left\{ 1 - \ln \left(\sum \theta_j \tau_{ji}\right) - \sum_{j=1}^{nc} \frac{\theta_j \tau_{ij}}{\sum\limits_{k=1}^{nc} \theta_k \tau_{kj}} \right\} \tag{3.25}$$

where

$$l_i = \frac{z}{2}(r_i - q_i) - (r_i - 1) \tag{3.26}$$

$$\theta_i = \frac{q_i x_i}{\sum\limits_{j=1}^{nc} q_j x_j} \tag{3.27}$$

$$\phi = \frac{r_i}{\sum\limits_{j=1}^{nc} r_j x_j} \tag{3.28}$$

$$v_i = \frac{q_i \sum\limits_{j=1}^{nc} r_j x_j}{r_i \sum\limits_{j=1}^{nc} q_j x_j} \tag{3.29}$$

$$\tau_{ij} = \exp\left(- \frac{u_{ij}}{RT} \right) \tag{3.30}$$

where the area and volume parameters, (r, q), and binary interaction parameters $u_{i,j}$ have been taken from Venimadhavan et al. (1999). The area and volume parameters are shown in Table 3.2 and binary interaction parameters are shown as in matrix form as.

$$u_{i,j} = \begin{bmatrix} 0 & -131.7686 & -298.434 & -343.593 \\ -148.2833 & 0 & 85.5336 & 68.0083 \\ 712.2349 & 24.6386 & 0 & 685.71 \\ 527.9269 & 581.1471 & 461.4747 & 0 \end{bmatrix}$$

The iterative procedure is repeated until the previous and current estimated tray temperatures become approximately equal. The values of vapor composition on the tray is estimated at the final iterative tray temperature. The procedure is repeated for all the trays.

3.1.3.3 Enthalpy Calculations

Enthalpy mentioned in the energy balance equations is a temperature and composition dependent entity. It can be estimated from the following equations.

$$H_i = \sum_{j=1}^{N_c} y_{i,j} H_{i,j}^V \tag{3.31}$$

$$h_i = \sum_{j=1}^{N_c} x_{i,j} H_{i,j}^L \tag{3.32}$$

where

$$H_{i,j}^V = \sum_{j=1}^{N_c} T_i C_{p_{i,j}}^V \tag{3.33}$$

$$H_{i,j}^L = \sum_{j=1}^{N_c} (H_{i,j}^V - H_{i,j}^{latent}) \tag{3.34}$$

where $C_{p_{i,j}}^V$ is heat capacity of the gas in $J\ mol^{-1} K^{-1}$, which is temperature dependent and can be estimated as.

$$C_{p_{i,j}}^V = A_j + B_j T_i + C_j T_i^2; \tag{3.35}$$

The $H_{i,j}^{latent}$ in Eq. 3.34 is a latent heat of vaporization, which can be estimated from Watson's equation as.

$$H_{T_1}^{latent} = H_{T_2}^{latent} \left(\frac{1 - Tr_1}{1 - Tr_2} \right)^{0.38} \tag{3.36}$$

where

$$T_{r_1} = \frac{T_j}{T_{c_j}} \tag{3.37}$$

$$T_{r_2} = \frac{Tb_j}{T_{c_j}} \tag{3.38}$$

where T_{b_i} and T_{c_i} are boiling and critical temperature of the compound j. All the specific heat constants and compounds properties have been taken from Yaws (1999) and are shown in Table. 3.3.

3.1.4 Liquid-Liquid Equilibrium

As the column is having a decanter for the phase separation from the condenser outlet, the decanter needs to be modeled. The concept of liquid-liquid equilibrium (LLE) has

Table 3.3: Heat capacity constants and physical properties of the compounds

	A	B	C	Tb (K)	Tc (K)	$H_{Tb}(Jmol^{-1})$
Acetic Acid	-422.58	-4.83×10^{-2}	2.33×10^{-5}	390.9	594.25	23697
Butanol	-245.80	-1.123×10^{-1}	5.35×10^{-5}	390.7	562.75	43124
Butyl Acetate	-451.56	-1.35×10^{-1}	6.99×10^{-5}	399.7	578.85	36006
Water	-238.51	-1.23×10^{-2}	2.77×10^{-6}	373	646.15	40683

being applied to model the decanter. The LLE is applied when two liquid phases at certain composition are in thermal equilibrium with each other. If two liquid phases denoted by superscript, I and II, are in equilibrium with each other then.

$$\gamma_j^I x_j^I = \gamma_j^{II} x_j^{II} \tag{3.39}$$

$$x_j^I = K_j x_j^{II} \qquad \left(K_j = \frac{\gamma_j^{II}}{\gamma_j^I} \right) \tag{3.40}$$

In the case of decanter in RDC, there are two phases, organic and aqueous, let organic phase be denoted by superscript I and aqueous is II. Applying component material balance in decanter,

$$F z_j = x_j^I L^I + x_j^{II} L^{II} \tag{3.41}$$

$$z_j = x_j^I \alpha + x_j^{II} \alpha \qquad \left(\alpha = \frac{L^I}{F},\ 1 - \alpha = \frac{L^{II}}{F} \right) \tag{3.42}$$

where F is a outlet stream flow rate of condenser with z_j composition and the stream is going as an inlet into the decanter. α is a split fraction which can be estimated by solving the above equation as.

$$x_j^I = \frac{z_j}{\alpha + (1-\alpha)K_j} \tag{3.43}$$

Since,

$$\sum_{j=1}^{N_C} x_j^I = x_j^{II} = 1 \tag{3.44}$$

The Eq. 3.43 can also be written as

$$\sum_{j=1}^{N_C} x_j^I = \sum_{j=1}^{N_C} \frac{z_j}{\alpha + (1-\alpha)K_j} = 1 \tag{3.45}$$

The above equation is solved using Newton Raphson's method to estimate the split fraction and the two phases composition. The separated phases are than used as reflux and distillate stream respectively.

3.2 Model Validation

A dynamic MATLAB® and SIMULINK® models are developed using above modeling equations. The model has been simulated using ODE15s solver which is a ordinary differential equation solver of MATLAB® software. The model is only considered legitimate if it is validated with some other trusted source. In this study, the model has been validated with a CHEMCAD® based simulated model and with an experimental study performed by Steinigeweg (2002). The model inputs for both the model are shown in Table 3.4.

Table 3.4: Model Specifications

	CHEMCAD®	Steinigeweg
Total Stages	15	28
Feed Stages	6 & 9	7 & 11
Reactive Stages	6–9	6-22
P_1 (kPa)	101.312	102.646
δP (kPa)	0.015	0.771
F_{HOAc} (mol/h)	10.5	23
F_{BuOH} (mol/h)	9.5	52
Q_B (kW)	0.972	1.29

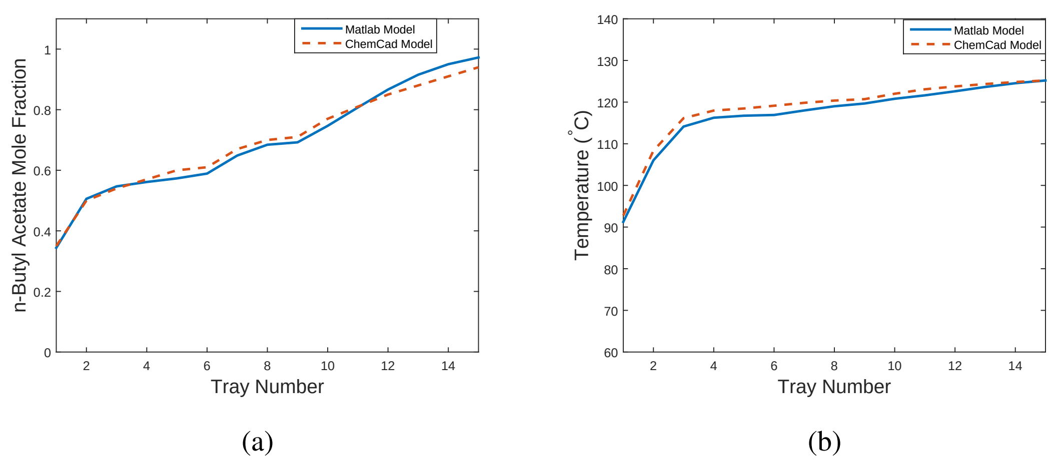


Figure 3.3: Comparison between MATLAB® and CHEMCAD® Models (a) Composition Profile (b) Tray Temperature Profile

3.2.1 Validation with CHEMCAD® Simulated Model

The CHEMCAD® and MATLAB® models are compared to each other on the basis of *n*- Butyl Acetate composition and tray temperature profile. The reason of using these factors is that these two are the prime variables which are used in soft sensor and control studies. The comparison of composition and tray temperature profile between CHEMCAD® and MATLAB® is shown in Fig. 3.3. The R^2 values of compostion and tary temperature profiles are 0.989 and 0.995 respectively. It can be concluded from the profiles and R^2 values that MATLAB® model is approximately fitting with CHEMCAD® simulation model and can be used for the future studies.

Table 3.5: Comparison between Matlab Model and Experimental Product Values

	MATLAB® Model	Experimental Study
D (mol/h)	23.25	24
x^D_{HoAc}	0.127	0.121
x^D_{BuOL}	0.003	0.006
x^D_{BuAc}	0.009	0.002
x^D_{Water}	0.861	0.87
B (mol/h)	51.74	51
x^B_{HoAc}	0.005	0.001
x^B_{BuOL}	0.618	0.586
x^B_{BuAc}	0.382	0.381
x^B_{Water}	0.005	0.003

3.2.2 Validation with Experimental Studies

A model is not considered accurate until it is not giving output close to the experimental values. The MATLAB® model is also validated with the experimental values mentioned in Steinigeweg (2002). The composition profile of all the components has been shown in Fig. 3.4. It can be observed from the composition profile that butanol profile is fitting accurately with the experimental values. In the case of butyl acetate, some deflection can be seen in the reactive tray section. The reason might be because of unknown loading of catalyst across the trays and some deflection in reaction kinetics taken theoretically from experimental kinetics. The top and bottom product properties are also compared between the model and experimental study as shown in Table. 3.5. It can be observed that even there was some deviation in the column composition profile, the top and bottom products properties are still comparably close between the MATLAB® model and experimental values.

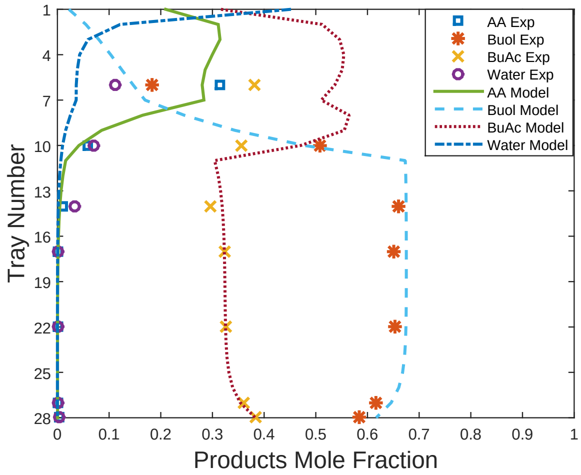


Figure 3.4: Comparison between MATLAB® model and Steinigeweg (2002) Experimental Study

Since the profiles of MATLAB® model is showing satisfying fitting with both CHEMCAD® simulated model and experimental values, it can be used for the future sensor and controller studies.

3.3 Open Loop Response

To study the dynamic characteristics of the RDC, MATLAB® model is converted to SIMULINK® based dynamic model. It involved development of an S-function and transforming the S-function into an open loop as shown in Fig. 3.5. The open loop of the RDC contains three inputs, involving two reactant feeds and one reboiler duty. The loop is having 31 outputs involving 15 tray temperatures, 15 state values of tray holdups and lastly the *n*- Butyl Actetate composition in the bottoms product. For the control purpose two reactant feeds are considered as load variables and reboiler duty is considered as a input variable of the model. The open loop response of the RDC model was studied by adding certain disturbances in the load variables and input variable to observe the dynamic changes in the product composition.

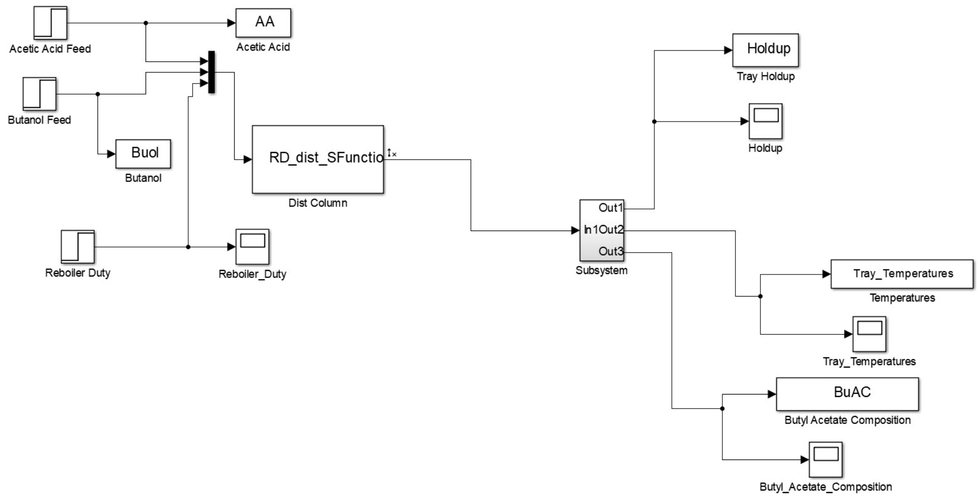


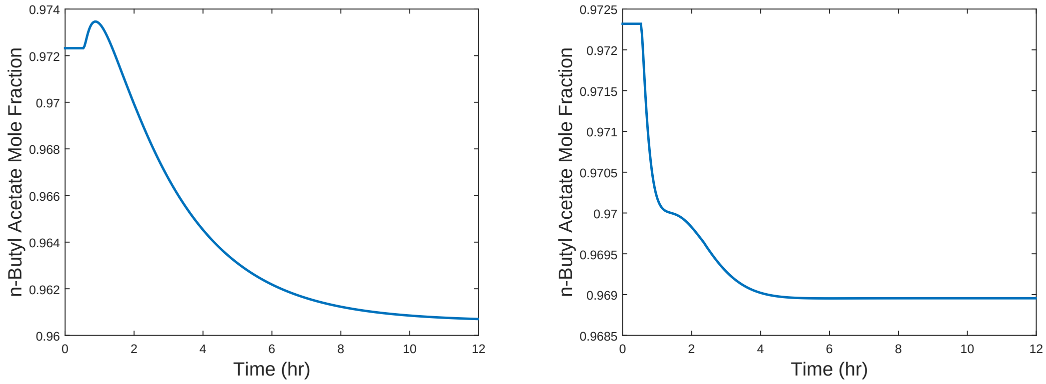
Figure 3.5: SIMULINK® Diagram of RDC Open Loop

3.3.1 Load Change in Acetic Acid Feed Rate

The model works at steady state with butyl acetate mole fraction of 0.9723 in bottoms stream at acetic acid feed rate of 10.5 $kmol/hr$. The open loop response was studied by giving a step change in the feed rate of acetic acid by $\pm 10\%$. As shown in Fig. 3.6(a), the process is showing inverse response for the step change of -10%. The inverse response is because of the reaction and separation processes occurring at the same time. When acetic acid feed rate is decreased it leads to decrease in the heavier compound in the bottoms causing increase in the mole fraction of butyl acetate at the starting of process but after that it also leads to decrease in production of butyl acetate in the end. In the case of Fig.3.6 (b), giving positive change causes a high dosage of heavier compound at bottoms which leads to decrease in the mole fraction of butyl acetate. This inverse response causes complexity in designing the control system for this RDC column. The problem of inverse response has been discussed in Chapter 4.

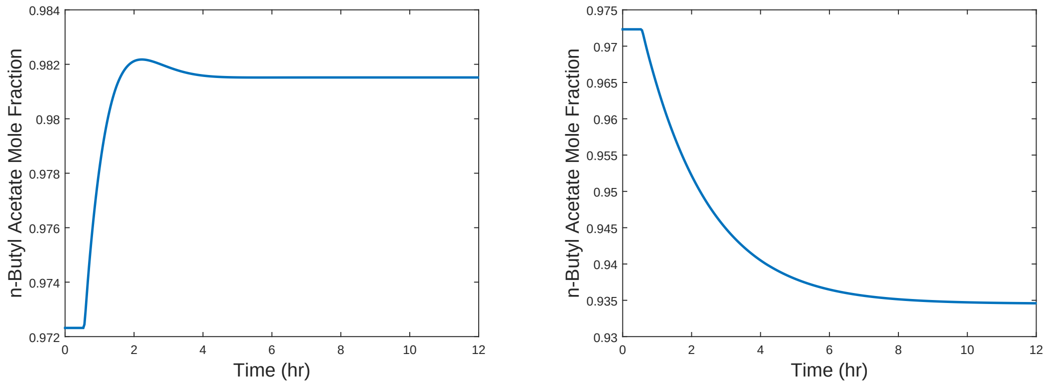
3.3.2 Load Change in Butanol Feed Rate

The open loop response was also studied by giving a disturbance in second load variable of the model. Step changes of $\pm 10\%$ were given to the feed rate of butnaol and the change in dynamics were observed as shown in Fig. 3.7. The decrease in butanol feed rate leads to the decrease in the quantity of unreacted butanol reaching the bottoms stream and increasing the mole fraction of butyl acetate in the product stream. Similarly increase in the butanol feed leads to the opposite effect on the product stream.



(a) (b)

Figure 3.6: Open Loop Response for Step Change in Acetic Acid Feed Flow Rate by $(a) - 10\%$ $(b) 10\%$



(a) (b)

Figure 3.7: Open Loop Response for Step Change in Butanol Feed Flow Rate by $(a) - 10\%$ $(b) 10\%$

3.3.3 Step Change in Reboiler Duty

The reboiler duty being an input variable was also given a step change of $\pm 10\%$. The open loop response is shown in Fig.3.8. It can be seen that reboiler duty affects the process with a considerable change in product composition in both the positive and negative changes. The reboiler of RDC also works the same as conventional distillation column. By increasing the reboiler duty, the heavier product composition increases as it causes vaporization of the lighter components. Similarly decrease in reboiler duty decreases the product composition of butyl acetate in the product stream.

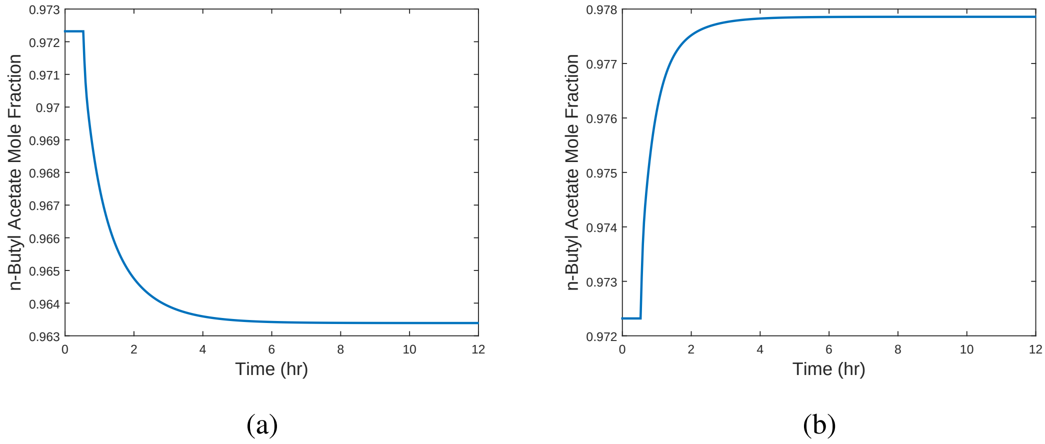


Figure 3.8: Open Loop Response for Step Change in Reboiler Duty by $(a) - 10\%$ $(b)10\%$

3.4 Conclusions

In this chapter, reactive distillation column carrying an esterification reaction for *n*-Butyl Acetate production has been studied. A model of RDC was proposed to undergo the future studies. The model contained various material, component and energy balances at every tray of the column. The methodologies to estimate various parameters like liquid and vapor enthalpies, liquid and vapor flow rates, condenser duty, etc. are also proposed for the modeling purpose. The RDC model was followed by the modeling of decanter which was needed after the condenser in the column. The proposed model was solved in the MATLAB® software which was validated sucesfully using CHEMCAD® software based simulation model and Steinigeweg (2002) experimental study. Finally after validation, the open loop response of the model was studied for the future control studies. It was observed that RDC was showing the problem of inverse response due to load variable which is encountered in the case of negative step change in the acetic acid feed rate. This inverse response can create certain problems in the control studies, so it needs special considerations in further studies.

Chapter 4

Inverse Response Behavior of Reactive Distillation Column

During inverse response, initially, the output of an open loop goes in the opposite direction to that direction where the response ends. The reason for the inverse response is when two opposing processes work simultaneously, and one process is dominating at the starting of the process but the second process prevails in the end. The inverse response has a characteristic of having atleast one zero which is a positive real number or imaginary pole with positive real part in the process transfer function (Iinoya and Altpeter, 1962). The inverse response in the process can occur during the step change given either in manipulated variable or in load variable. In the case of inverse response in the manipulated variable, the problem can be solved by compensating the process using a compenstor as mentioned in Stephanopoulos (1984). But in the case of inverse response due to step change in load variable, compensating the process using Smith Predictor is out of scope. This inverse response problem due to load variable was also encountered by Vijaya Raghavan et al. (2011) but remained untouched. The problem has to be handled using a controller having either feedforward (FF) properties or having future prediction characteristics.

In this chapter, the problem of inverse response due to both load variable and manipulating variable has been discussed. In the case of load variable, a feedforward-feedback (FF-FB) controller has been designed for the problem of inverse response. The inverse response due to manipulated variable has been discussed by designing a Smith Predictor for the process having a 3^{rd} order transfer function.

4.1 Inverse Response due to Load Variable

In this study, it has been observed in Fig. 3.6 (a) that the process is showing inverse response during negative step change in acetic acid feed rate which is a load variable.

Since the inverse response is due to the step change in a load variable, it has to be removed from the system before it can enter the plant. This leads to the use of feedforward controlling technique for the removal of inverse response in the process.

4.1.1 Feedforward-Feedback Control

Let us take a process, $G_p(s)$, with two load variables, $G_{d_1}(s)$ and $G_{d_2}(s)$, and one manipulated variable, m. The open loop of the process has been shown in Fig. 4.1. The output of the process, $\bar{y}(s)$, can be controlled by a feedback controller by manipulating the variable m . But to solve the problem of inverse response due to a load variable,

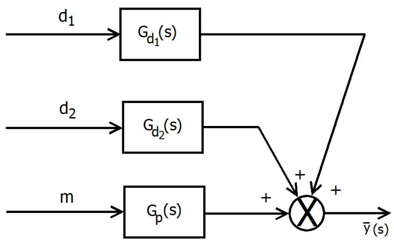


Figure 4.1: Open loop of the process

a feedforward controller coupled with feedback controller is needed for the removal of disturbances, d_1 and d_2, before they can enter the plant. The closed loop with FF-FB controllers is shown in Fig. 4.2. The output of the closed loop can be estimated as

$$\bar{y} = mG_p + d_1 G_{d_1} + d_2 G_{d_2} \tag{4.1}$$

$$\bar{y}(s) = y_{sp_1} G_{c_1} G_p + y_{sp_2} G_{c_2} G_p + d_1(G_{d_1} - G_{c_1} G_p) + d_2(G_{d_2} - G_{c_2} G_p) \tag{4.2}$$

To design the feedforward controllers, disturbances are rejected and the coefficients of d_1 and d_2 are equated to zero.

$$G_{c_1} = \frac{G_{d_1}}{G_p} \tag{4.3}$$

$$G_{c_2} = \frac{G_{d_2}}{G_p} \tag{4.4}$$

In the case of feedback controller, a PID controller is used which can be tuned using techniques such as Ziegler-Nicholas, Cohen-Coon etc.

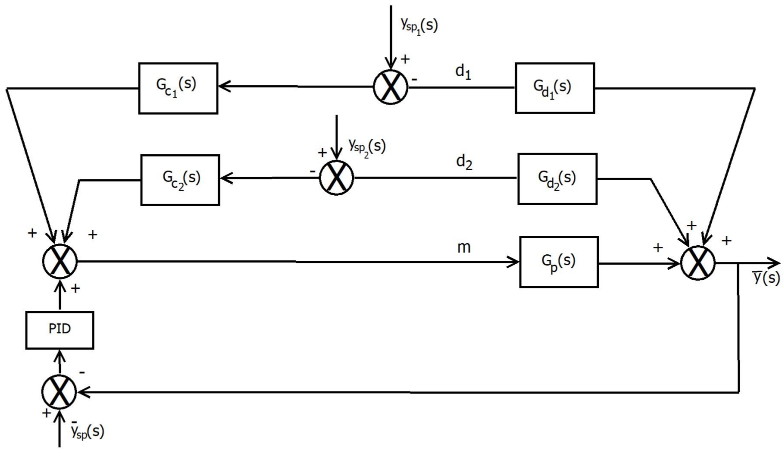


Figure 4.2: Closed loop having Feedforward-Feedback Controllers

4.1.2 Controller Performance

The effect of FF controller is compared by using closed loop with just FB controller and with FF-FB controller separately. The performance of the both the FB and FF-FB controllers needs to be evaluated on some error criteria. The integral errors such as integral of square of error (ISE), integral of the absolute value of error (IAE) and integral of time-weighted absolute error (ITAE) are used for the purpose of evaluating controllers performance (Stephanopoulos, 1984). The ISE criteria is used when the value of error is large, IAE is used in the case of small errors and ITAE is used when error is persistent over the time. If e is the difference between set point and the process output at time t, then integral errors can be estimated as.

$$ISE = \int_0^\infty e^2\, dt \tag{4.5}$$

$$IAE = \int_0^\infty |e|\, dt \tag{4.6}$$

$$ITAE = \int_0^\infty |e|\, t\, dt \tag{4.7}$$

4.1.3 Transfer Functions

As feedforward-feedback controller is a model dependent controller, transfer functions of G_P, G_{d_1} and G_{d_2} needs to be estimated for the estimation of controllers transfer functions. The transfer functions of process and both the disturbances are estimated using data obtained by giving a step change in manipulated variable and both the load variables. The estimated transfer function are shown as.

$$G_p = \frac{0.077s^2 + 17.46s + 27.25}{s^3 + 78.11s^2 + 2116s + 2851} \tag{4.8}$$

$$G_{d_1} = \frac{-2.73 \times 10^{-03}s^2 - 0.132s + 0.13}{s^3 + 19.38s^2 + 115.35s + 49.05} \tag{4.9}$$

$$G_{d_2} = \frac{6.77 \times 10^{-03}s^2 - 0.549s - 3.045}{s^3 + 53.25s^2 + 536s + 944.6} \tag{4.10}$$

The percent fit and mean square error between RDC SIMULINK® model and transfer function model are given in Table. 4.1. From these transfer functions the FF controllers are estimated using Eq. 4.3 and 4.4 as.

$$G_{c_1} = \frac{-2.72 \times 10^{-03}s^5 - 0.35s^4 - 6.67s^3 + 1.93s^2 + 237.24s + 370.34}{7.71 \times 10^{-02}s^5 + 18.95s^4 + 374.52s^3 + 2545.88s^2 + 3999.7s + 1336.61} \tag{4.11}$$

$$G_{c_2} = \frac{6.77 \times 10^{-03}s^5 - 0.02s^4 - 31.6s^3 - 1380.22s^2 - 8008.4s - 8681.29}{7.71 \times 10^{-02}s^5 + 21.57s^4 + 998.35s^3 + 10882.5s^2 + 31098.7s + 25740.3} \tag{4.12}$$

The PID controller for the feedback loop has been tuned using MATLAB® automatic

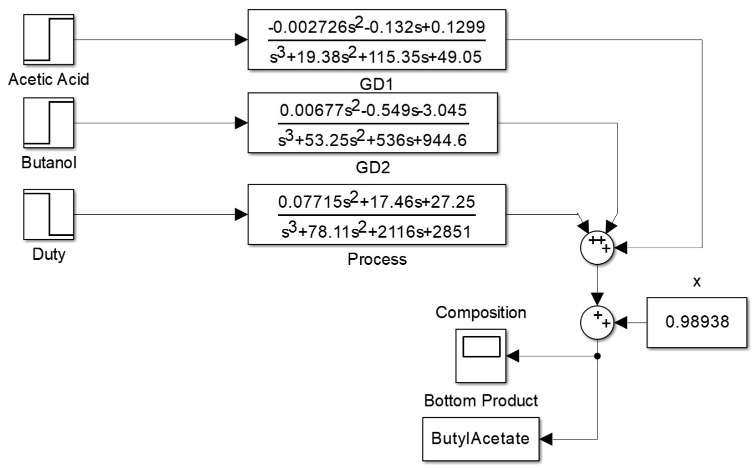


Figure 4.3: SIMULINK® Model of Open loop of the Process

tuning with the PID parameters as P = 166.27, I = 112.6 min^{-1} and D = 18.4 min. The MATLAB® auto tuning technique works on the principle of tuning the PID gains by choosing the crossover frequency according to the plant dynamics and designs for the phase margin with the target value of $60°$.

Table 4.1: Percent Fit and MSE between RDC model and Transfer Function Model

Transfer function	R^2	MSE
G_p	99.03	1.58×10^{-12}
G_{d_1}	99.14	4.18×10^{-11}
G_{d_2}	99.56	2.52×10^{-12}

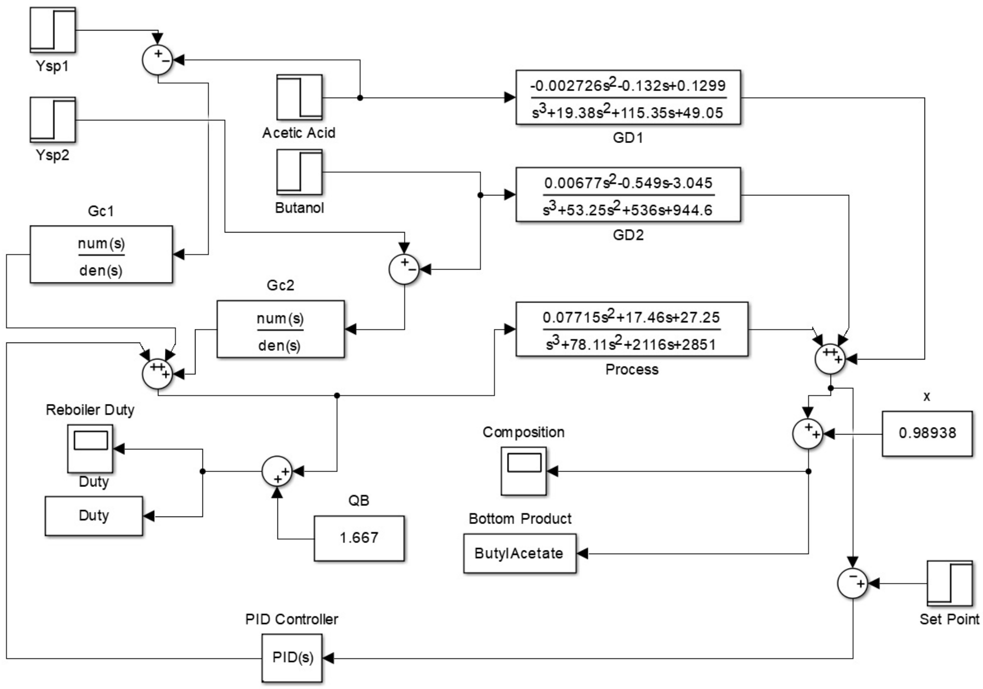


Figure 4.4: SIMULINK® Model of Closed loop having Feedforward-Feedback Controllers

4.1.4 Control Loops

Using the above mentioned transfer functions, the open loop and closed loop models are developed in SIMULINK® software as shown in Fig. 4.3 and 4.4. The open and closed loop dynamics were studied by giving step changes in load and manipulating variables. Target of the closed loop is to maintain the composition of *n*-butyl acetate at the value of 0.98938.

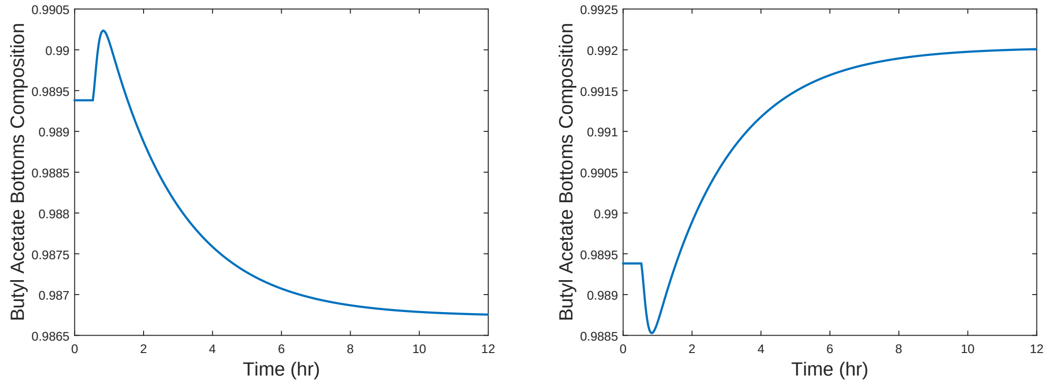


Figure 4.5: Open Loop Response for Step Change in Acetic Acid Feed Flow Rate by (a) -10% (b) 10%

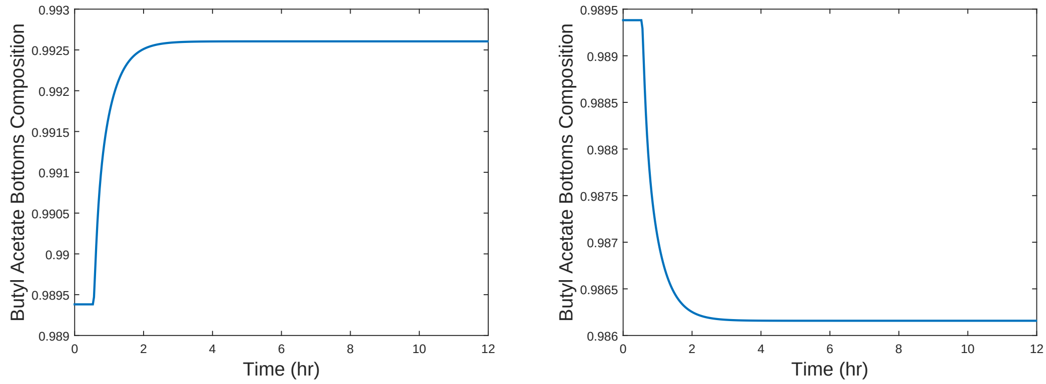


Figure 4.6: Open Loop Response for Step Change in Butanol Feed Flow Rate by (a) -10% (b) 10%

4.1.5 Open Loop Response

The step change was given in both the load variables and its behavior was studied for the control purpose. The open loop response, when step change of $\pm 10\%$ is given in acetic acid feed flow rate and butnaol are shown in Fig. 4.5 and 4.6. It can be noticed that open loop response during the step change in acetic acid is showing an inverse response. The transfer function just reverses the response during opposite step change and produces inverse response at positive change also. This is the problem of using transfer function that it does not show the exact non linearity of the plant at every step change. In the case of load change in butanol feed rate, the response is comparably similar to Fig. 3.7, which is showing opposite deflections in the product composition response on opposite step changes in the butanol composition.

4.1.6 Closed Loop Response

The bottoms composition of butyl acetate was controlled using closed loop with FB and FF-FB controllers. As adding a feedforward controller does not affect the servo problem of the closed loop, so only regulatory problem in the loop was studied.

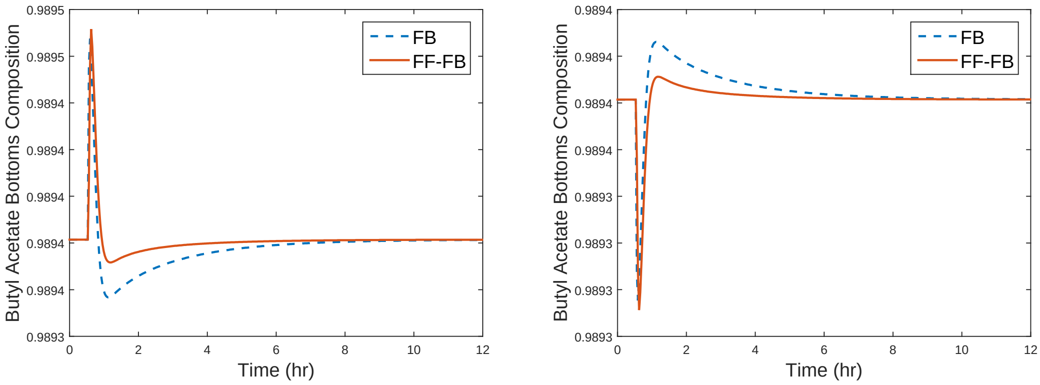


Figure 4.7: Closed Loop Response for Step Change in Acetic Acid Feed Flow Rate by (a) -10% (b) 10%

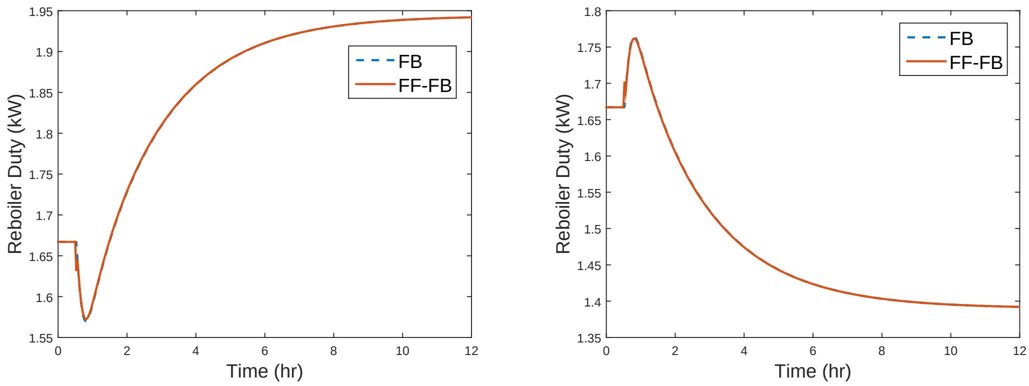


Figure 4.8: Reboiler Duty during Step Change in Acetic Acid Feed Flow Rate by (a) -10% (b) 10%

4.1.6.1 Load Change in Acetic Acid Feed Flow Rate

The closed loop response of the process for step change of $\pm 10\%$ in the feed flow rate of acetic acid is shown in Fig. 4.7 and its change on the manipulative variable is shown in Fig. 4.8. It can be observed that FF-FB controller is showing better control results than FB controller and removing the sluggishness problem of the process by controlling the composition 2-2.5 hr earlier than its counterpart. The integral errors for both the closed loop responses are shown in Table. 4.2 and it can be seen that integral errors are decreasing while using FF-FB controller.

Table 4.2: Integral Errors during Load Change in Acetic Acid Feed Flow Rate

	-10%		10%	
	FB	FF-FB	FB	FF-FB
ISE	1.62×10^{-09}	1.18×10^{-09}	1.62×10^{-09}	1.18×10^{-09}
IAE	6.90×10^{-05}	3.43×10^{-05}	6.90×10^{-05}	3.43×10^{-05}
ITAE	1.34×10^{-04}	4.17×10^{-05}	1.34×10^{-04}	4.17×10^{-05}

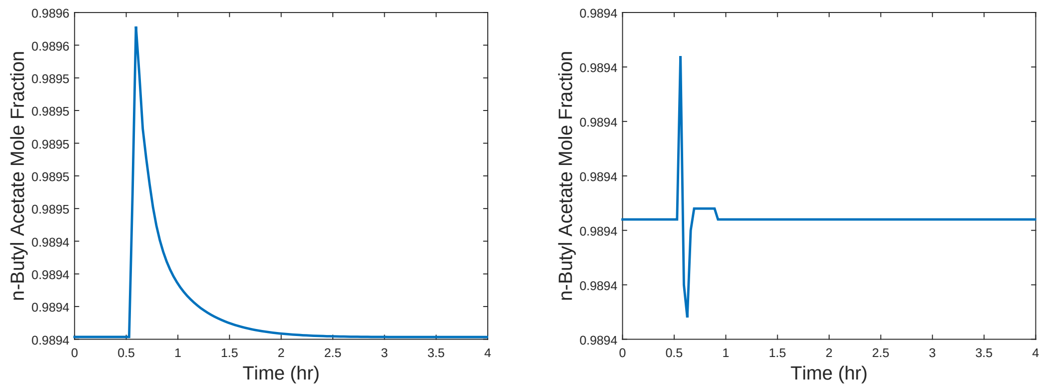


Figure 4.9: Closed Loop Response for -10% Step Change in Butanol Feed Flow Rate havinng (a) FB Controller (b) FF-FB Controller

4.1.6.2 Load Change in Butanol Feed Flow Rate

The closed loop response for the second load variable is studied by giving a step change of $\pm 10\%$ in feed flow rate of butanol. The closed loop response is shown in Fig. 4.9 and 4.10 and change in reboiler duty after the step change is shown in Fig. 4.11. While FB controller was controlling the output in 1.5-2 hr, FF-FB is controlling the same output in maximum half hour. The integral errors as shown in Table. 4.3 are also getting decreased to high extent when FF-FB controller is used.

4.1.6.3 Process Transfer Function Mismatch

The process model robustness was checked by changing the coefficient of s^3 of the characteristic equation by $\pm 50\%$ and then studying the regulatory problem. A step

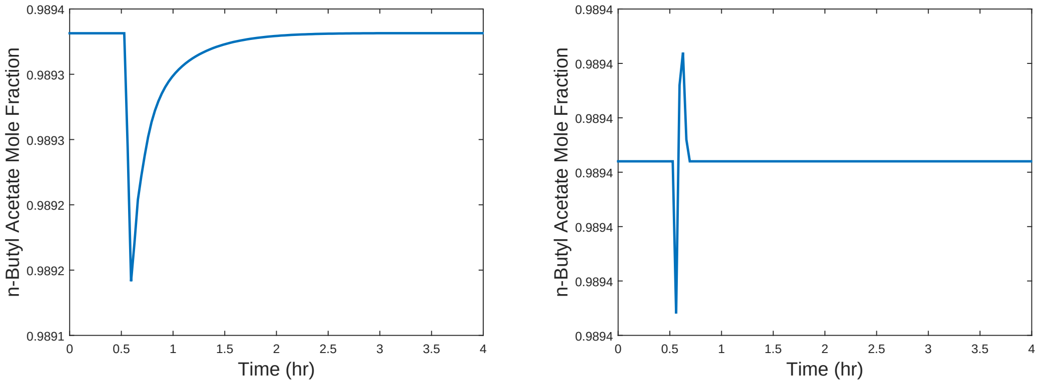


Figure 4.10: Closed Loop Response for 10% Step Change in Butanol Feed Flow Rate havinng (a) FB Controller (b) FF-FB Controller

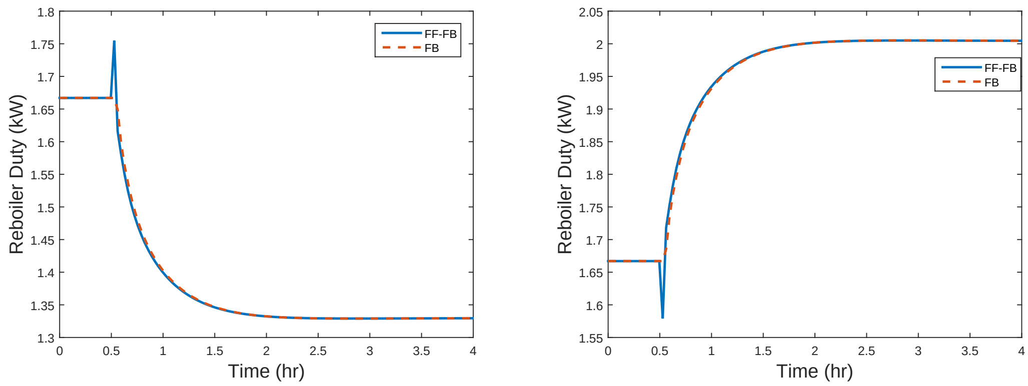


Figure 4.11: Reboiler Duty during Step Change in Butanol Feed Flow Rate by (a) -10% (b) 10%

change of -10% was given in both the feed flow rates individually, and the response was observed as shown in Fig. 4.12. During step change in acetic acid flow rate, the change in coefficient does not affect the process much. The step change in butanol feed flow rate is affecting the response to the higher extent. The response starts up with some oscillations, and finally output settles at its set point while taking some more time. The integral errors are shown in Table. 4.4 and it can be observed that errors are not changing much during acetic acid feed rate change but a high change can be observed during a step change in butanol feed rate.

Table 4.3: Integral Errors during Load Change in Butanol Feed Flow Rate

	-10%		10%	
	FB	FF-FB	FB	FF-FB
ISE	4.47×10^{-09}	1.27×10^{-17}	4.47×10^{-09}	1.28×10^{-17}
IAE	5.07×10^{-05}	5.24×10^{-09}	5.07×10^{-05}	5.24×10^{-09}
ITAE	1.71×10^{-05}	2.42×10^{-08}	1.71×10^{-05}	2.42×10^{-08}

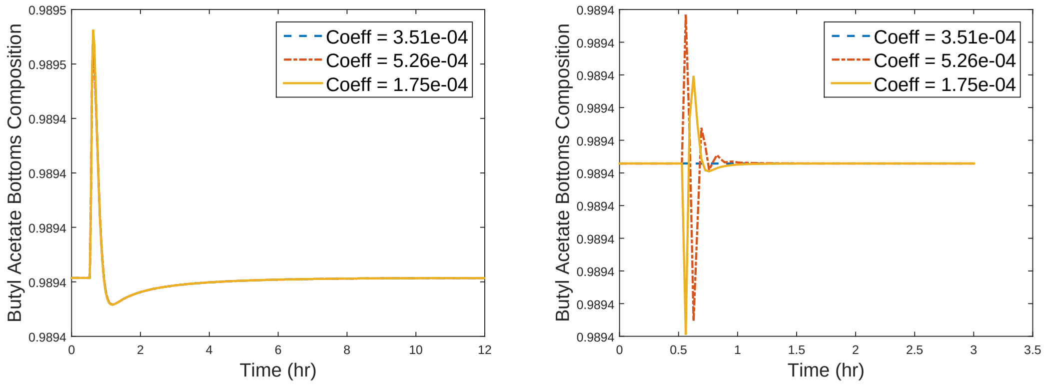


Figure 4.12: Process model mismatch by $\pm 50\%$ (a) -10% step change in Acetic Acid Feed Rate (b) -10% step change in Butanol Feed Rate

4.1.6.4 Disturbances Transfer Function Mismatch

The model robustness has also been tested by changing the transfer function coefficients of s^3 in the characteristic equation of both the disturbances. The response of the closed loop was noticed by giving a step change of -10% in acetic acid feed rate when GD_1 coefficient is given a change of $\pm 50\%$ and -10% step change in butanol feed rate when coefficient of GD_2 was changed by $\pm 50\%$. The closed loop response is shown in Fig. 4.13. The mismatch in G_{D_1} coefficient is not affecting the process much. In the case of G_{D_2} coefficient mismatch, a considerable difference in closed loop response is observed but still the response is staying within the manageable limits. The integral errors are shown in Table. 4.5. The integral errors are not changing much during G_{D_1} coefficient change but G_{D_2} coefficient change is showing noticeable change in integral errors but in the ignorable limits.

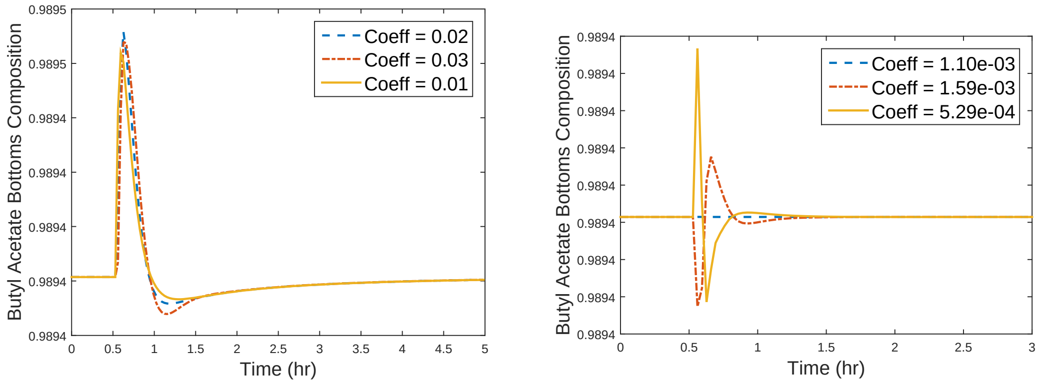


Figure 4.13: Disturbances model mismatch (a) $\pm 50\%$ mismatch in GD_1 (b) $\pm 50\%$ mismatch in GD_2

Table 4.4: Integral Errors During Process Model Mismatch

	Step Change in Acetic Acid Feed Rate			Step Change in Butanol Feed Rate		
Coeff. Value	3.51×10^{-04}	5.26×10^{-04}	1.75×10^{-04}	3.51×10^{-04}	1.75×10^{-04}	5.26×10^{-04}
ISE	1.18×10^{-09}	1.22×10^{-09}	1.13×10^{-09}	1.95×10^{-17}	3.16×10^{-11}	4.11×10^{-11}
IAE	3.43×10^{-05}	3.46×10^{-05}	3.42×10^{-05}	6.03×10^{-09}	1.98×10^{-06}	2.43×10^{-06}
ITAE	4.17×10^{-05}	4.17×10^{-05}	4.16×10^{-05}	2.48×10^{-08}	2.22×10^{-07}	2.89×10^{-07}

4.1.7 Feedforward-Feedback Controller for Plant Model

The feedforward-feedback controller proved to be a appropriate option for the removal of inverse response that is present in the process because of step change in load variable. The controller provided excellent results when the process is a represented as a transfer function model. The controller also need to be tested when the actual plant or non linear S-function model is considered for the problem. The reason behind this validation is that, the transfer function model carries very limited knowledge about the process and the controller obtained has to be validated at every situation possible.

4.1.7.1 Closed Loop

The controllers obtained using Eq. 4.3 and 4.4 are used in the closed loop of S-function model of the process as shown in the Fig. 4.14. In the closed loop, the reboiler duty which is a manipulated variable, is coming out from PID controllers, G_{C_1} and G_{C_2} outputs. The open loop response of the model has already been discussed in Section 3.3. The closed loop dynamics of the model can be studied as,

Table 4.5: Integral Errors During Model Mismatch of GD_1 and GD_2

	Change in GD_1 coefficient			Change in GD_2 coefficient		
Coeff.	0.02	0.03	0.01	1.10×10^{-03}	1.59×10^{-03}	5.29×10^{-04}
ISE	1.18×10^{-09}	1.01×10^{-09}	1.31×10^{-09}	1.95×10^{-17}	9.48×10^{-11}	5.14×10^{-11}
IAE	3.43×10^{-05}	3.23×10^{-05}	3.65×10^{-05}	6.03×10^{-09}	3.75×10^{-06}	3.68×10^{-06}
ITAE	4.17×10^{-05}	4.09×10^{-05}	4.27×10^{-05}	2.48×10^{-08}	5.61×10^{-07}	6.58×10^{-07}

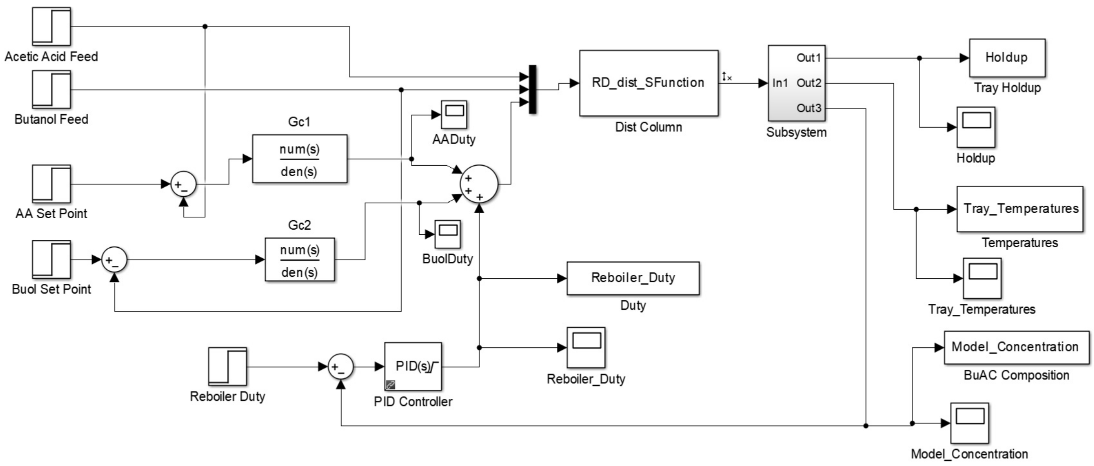


Figure 4.14: SIMULINK® Model of Closed loop having S-function Model of the Plant with Feedforward-Feedback Controllers

4.1.7.2 Load Change in Acetic Acid Feed Flow Rate

The closed loop response of the process when step change of -8% and $+10\%$ is given in the feed flow rate of acetic acid is shown in Fig. 4.7 and its change on the manipulated variable is shown in Fig. 4.8. It can be observed that FF-FB controller is showing better control results than FB controller during the negative change of acetic acid feed rate. The process is showing more overshoot and sluggishness during the positive change in acetic acid feed rate. The positive response results are not satisfactory because the transfer function of the G_{D_1} was estimated by giving the negative step change in acetic acid feed rate and thus not covering the dynamics of positive change accurately. This is the reason that the controller is operating better for the negative step changes but it cannot cover the positive changes appropriately. It can be observed from Fig. 3.6 that both the responses are in the same decreasing direction. If the direction could have been opposite, then the controllers might have acted in a positive manner. The integral errors

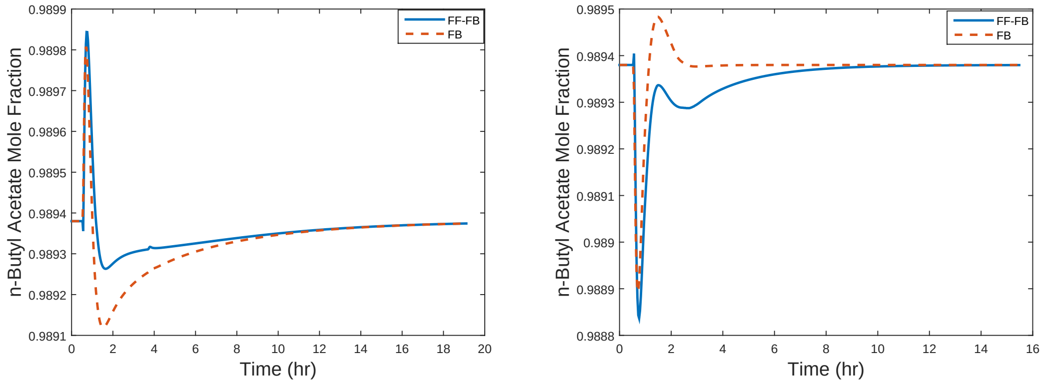


Figure 4.15: Closed Loop Response for Step Change in Acetic Acid Feed Flow Rate by (a) -8% (b) 10%

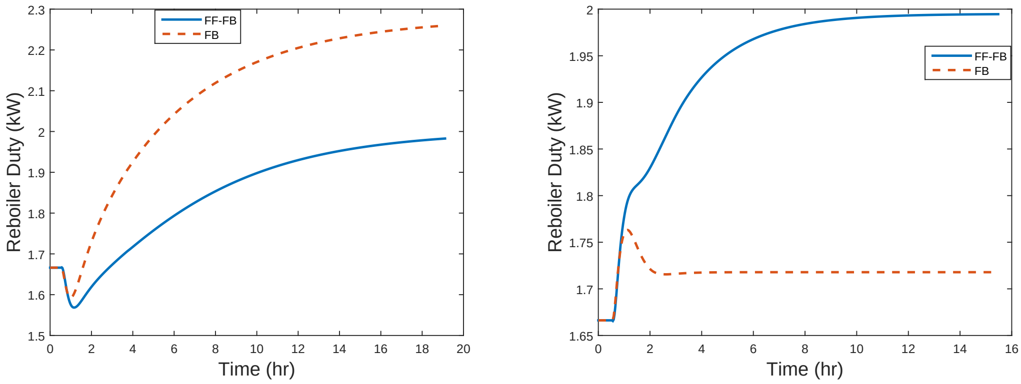


Figure 4.16: Reboiler Duty during Step Change in Acetic Acid Feed Flow Rate by (a) -8% (b) 10%

for both the closed loop responses are shown in Table. 4.15 . It can be seen that during the use of FF-FB controllers, integral errors are decreasing for the negative step change but increasing in the case of positive step change in the acetic acid feed rate.

4.1.7.3 Load Change in Butanol Feed Flow Rate

The regulatory problem for the second load variable is studied by giving a step change of $+8\%$ and -10% in feed flow rate of butanol. The closed loop response is shown in Fig. 4.17 and change in the reboiler duty after the step change is shown in Fig. 4.18. Since the dynamics of the G_{D_2} are in opposite direction with the opposite step change as shown in Fig. 3.7, the transfer function is carrying adequate knowledge of non linearity of the model for the current situation. This is the reason that FFFB controller

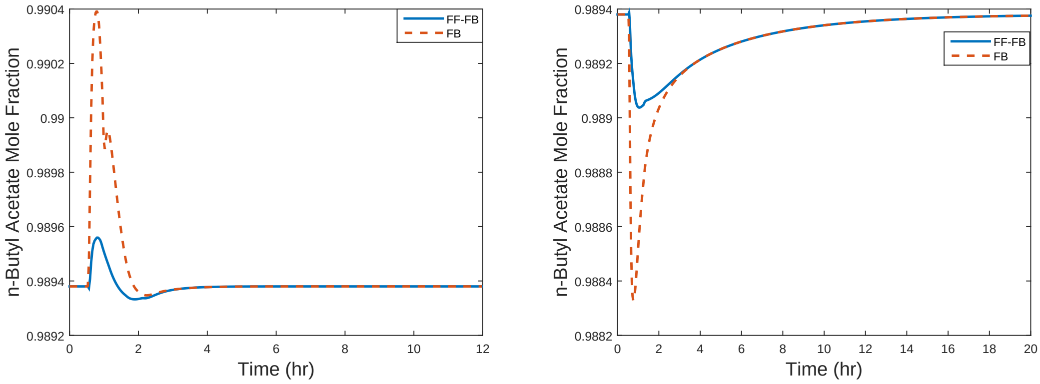


Figure 4.17: Closed Loop Response for Step Change in Acetic Acid Feed Flow Rate by (a) -10% (b) 8%

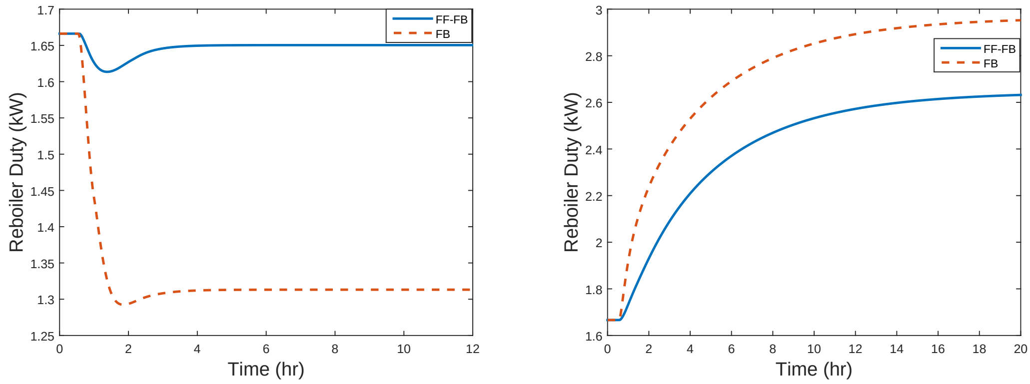


Figure 4.18: Reboiler Duty during Step Change in Acetic Acid Feed Flow Rate by (a) -10% (b) 8%

is performing excellently for both the positive and negative step changes in the butanol feed rate. The integral errors as shown in Table. 4.17 are also getting decreased to a good extent when FF-FB controller is used.

4.2 Inverse Response due to Manipulated Variable

Inverse response due to the manipulated variable is a common phenomenon which is experienced in the case of process operation units such as reboilers and boiler drums. This problem of inverse response is generally solved using a compensator which works on the principle similar to the Smith Predictor or by tuning a PID controller which concentrates on the positive zeroes of the transfer function. During the study of transfer function calculations and inverse response compensation, it was observed that in lit-

Table 4.6: Integral Errors during Load Change in Acetic Feed Flow Rate

	-8%		10%	
	FB	FF-FB	FB	FF-FB
ISE	1.71×10^{-07}	9.26×10^{-08}	6.14×10^{-08}	1.03×10^{-07}
IAE	1.18×10^{-03}	8.18×10^{-04}	2.33×10^{-04}	5.26×10^{-04}
ITAE	5.12×10^{-03}	4.01×10^{-03}	1.34×10^{-04}	1.10×10^{-03}

Table 4.7: Integral Errors during Load Change in Butanol Feed Flow Rate

	-10%		8%	
	FB	FF-FB	FB	FF-FB
ISE	4.08×10^{-07}	1.34×10^{-08}	8.24×10^{-07}	2.86×10^{-07}
IAE	6.30×10^{-04}	1.43×10^{-04}	2.05×10^{-03}	1.54×10^{-03}
ITAE	3.44×10^{-04}	1.36×10^{-04}	6.62×10^{-03}	6.35×10^{-03}

erature the compensator was limited to the 2^{nd} order process transfer function. It was already mentioned in Stephanopoulos (1984) and Iinoya and Altpeter (1962) that inverse response can also occur for the third order process. Using a case study, a compensator for 3^{rd} order transfer function model of the plant has been developed to remove the inverse response and dead time in the model.

4.2.1 Compensator for 3^{rd} Order Process

Assume that the following third order process without time delay is showing the inverse response.

$$G(s) = \frac{K(\tau s + 1)}{as^3 + bs^2 + cs + 1} \tag{4.13}$$

The process has atleast one zero which is positive or is a imaginary number with positive real part which is a cause of inverse response in the process output. This third order process is fragmented into two opposing processes having a first order process and a second order process using partial fractions.

$$G_1(s) = \frac{K_1(\tau s + 1)}{\tau_1 s^2 + 2\tau_1 \zeta s + 1} \tag{4.14}$$

$$G_2(s) = \frac{K_2}{\tau_2 s + 1} \tag{4.15}$$

where K_1 and K_2 are process gains, τ_1 and τ_2 are process time constants and ζ is a damping factor for second order process.

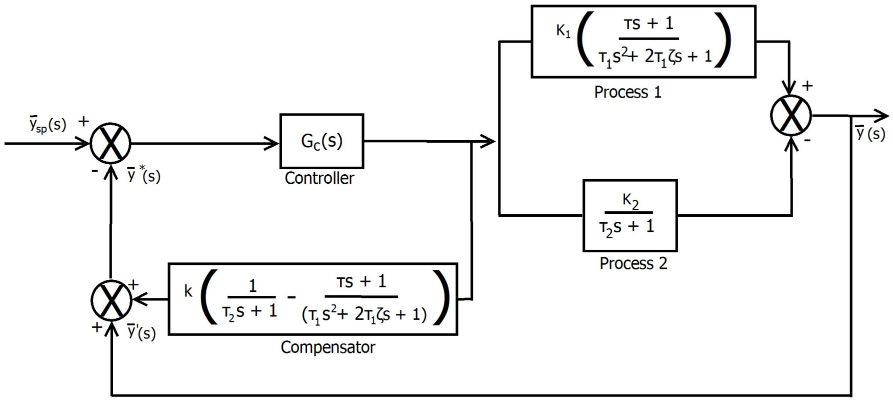


Figure 4.19: Closed loop with compensator

The overall open loop response of the process is

$$y(s) = G_c(s)\big(G_1(s) - G_2(s)\big)\bar{y}_{sp}(s) \tag{4.16}$$

where $G_c(s)$ is a transfer function of a controller and $\bar{y}_{sp}(s)$ is set point value of the required output.

$$y(s) = G_c(s)\frac{(K_1\tau\tau_2 - \tau_1^2 K_2)s^2 + (K_1(\tau + \tau_2) - 2K_2\tau_1\zeta)s + (K_1 - K_2)}{(\tau_1^2 s^2 + 2\tau_1\zeta s + 1)(\tau_2 s + 1)}\bar{y}_{sp}(s) \tag{4.17}$$

The roots of the numerator of the above equation are positive which are causing the problem of inverse response in the process. To eliminate the positive zeroes of the process, a compensator is added as shown in Fig.4.19.

$$y'(s) = G_c(s)k\left(\frac{1}{\tau_2 s + 1} - \frac{\tau s + 1}{\tau_1 s^2 + 2\tau_1\zeta s + 1}\right)\bar{y}_{sp}(s) \tag{4.18}$$

where k is a compensator gain. After adding compensator, the overall open loop response changes to

$$y^*(s) = y(s) + y'(s) \tag{4.19}$$

$$y^*(s) = G_c(s)\frac{(K_1\tau\tau_2 - K_2\tau_1^2 + k(\tau_1^2 - \tau\tau_2))s^2 + (K_1(\tau + \tau_2) - 2K_2\tau_1\zeta + k(2\tau_1\zeta - (\tau + \tau_2))s + (K_1 - K_2)}{(\tau_1^2 s^2 + 2\tau_1\zeta s + 1)(\tau_2 s + 1)}\bar{y}_{sp}(s) \tag{4.20}$$

To eliminate the inverse response of the process, the k value has to be optimized in such a way that zeroes of the transfer function,$y^*(s)$, lies on the negative left plane.
If α and β are the roots of the numerator (zeroes) of $y^*(s)$ then for the negative zeroes

$$\alpha\beta > 0 \quad and \quad \alpha + \beta < 0 \tag{4.21}$$

where

$$\alpha\beta = \frac{K_1 - K_2}{K_1\tau\tau_2 - K_2\tau_1^2 + k(\tau_1^2 - \tau\tau_2)} \tag{4.22}$$

$$\alpha + \beta = -\frac{K_1(\tau + \tau_2) - 2K_2\tau_1\zeta + k(2\tau_1\zeta - (\tau + \tau_2)}{K_1\tau\tau_2 - K_2\tau_1^2 + k(\tau_1^2 - \tau\tau_2)} \tag{4.23}$$

This leads to the two cases, $K_1 > K_2$ and $K_1 < K_2$

1. Considering the first case, i.e. when second process is dominating in the starting and finally first process prevails. If $K_1 > K_2$ then according to Eq: 4.21 denominator of Eq: 4.22 has to be greater than zero and numerator of Eq: 4.23 has to be less than zero.

   $$K_1\tau\tau_2 - K_2\tau_1^2 + k(\tau_1^2 - \tau\tau_2) > 0 \tag{4.24}$$

   and

   $$-\left(K_1(\tau + \tau_2) - 2K_2\tau_1\zeta + k(2\tau_1\zeta - (\tau + \tau_2)\right) < 0 \tag{4.25}$$

   Since sign shift may occur because of the coefficients of k in both the equations, the further cases needs to be considered are
   if $\tau_1^2 > \tau\tau_2$

   $$k > \frac{K_2\tau_1^2 - K_1\tau\tau_2}{\tau_1^2 - \tau\tau_2} \tag{4.26}$$

   else

   $$k < \frac{K_2\tau_1^2 - K_1\tau\tau_2}{\tau_1^2 - \tau\tau_2} \tag{4.27}$$

   and, if $\tau + \tau_2 > 2\tau_1\zeta$

   $$k < \frac{K_1(\tau + \tau_2) - 2K_2\tau_1\zeta}{\tau + \tau_2 - 2\tau_1\zeta} \tag{4.28}$$

   else

   $$k > \frac{K_1(\tau + \tau_2) - 2K_2\tau_1\zeta}{\tau + \tau_2 - 2\tau_1\zeta} \tag{4.29}$$

2. In second case, first process dominates in the initial stage but second process prevail in the end i.e. $K_1 < K_2$. Then according to Eq: 4.21 denominator of Eq: 4.22 has to be less than zero and numerator of Eq: 4.23 has to be greater than zero.

   $$K_1\tau\tau_2 - K_2\tau_1^2 + k(\tau_1^2 - \tau\tau_2) < 0 \tag{4.30}$$

   and

   $$-\left(K_1(\tau + \tau_2) - 2K_2\tau_1\zeta + k(2\tau_1\zeta - (\tau + \tau_2)\right) > 0 \tag{4.31}$$

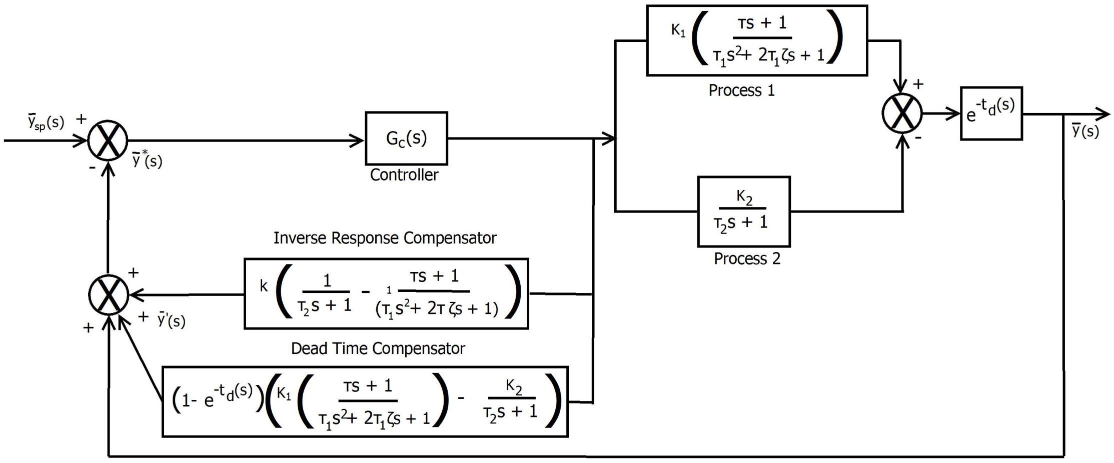


Figure 4.20: Process with Inverse Response and Dead Time Compensator

Again due to coefficients of k, sign shift can occur,so further cases can be considered as

if $\tau_1^2 > \tau\tau_2$

$$k < \frac{K_2\tau_1^2 - K_1\tau\tau_2}{\tau_1^2 - \tau\tau_2} \tag{4.32}$$

else

$$k > \frac{K_2\tau_1^2 - K_1\tau\tau_2}{\tau_1^2 - \tau\tau_2} \tag{4.33}$$

and, if $\tau + \tau_2 > 2\tau_1\zeta$

$$k > \frac{K_1(\tau + \tau_2) - 2K_2\tau_1\zeta}{\tau + \tau_2 - 2\tau_1\zeta} \tag{4.34}$$

else

$$k < \frac{K_1(\tau + \tau_2) - 2K_2\tau_1\zeta}{\tau + \tau_2 - 2\tau_1\zeta} \tag{4.35}$$

Using the optimized value of compensator gain, k, the zeroes of the process are converted from positive to negative roots. This technique can also be extended to third order processes with inverse response and a dead time, t_d.

$$G(s) = \frac{K(\tau s + 1)}{as^3 + bs^2 + cs + 1} e^{-t_d(s)} \tag{4.36}$$

The compensator as shown in Fig.4.20 is modified by removing dead time as well as inverse response factor of the process. The dead time compensator is a Smith predictor designed by removing the dead time factor from the overall response of the process.

$$y'(s) = G_c(s)\Big((1 - e^{-t_d(s)})\Big(\frac{K_1\tau s + 1}{\tau_1 s^2 + 2\tau_1\zeta s + 1} - \frac{K_2}{(\tau_2 s + 1)}\Big) + k\Big(\frac{1}{\tau_2 s + 1} - \frac{\tau s + 1}{\tau_1 s^2 + 2\tau_1\zeta s + 1}\Big)\Big)\bar{y}_{sp}(s) \tag{4.37}$$

The value of k can be optimized using the same method given above.

4.2.2 Case Study

The case study for studying the Smith predictor has been taken from Peng et al. (2003). They observed an inverse response in the product conversion in a reactive distillation column during -10% step change in a distillate flow rate. The data has been extracted from the figure and an approximate transfer function has been estimated using *system identification* toolbox of the MATLAB® software.

$$G_p(s) = \frac{0.1642s^2 - 51.23s + 48.24}{s^3 + 26.39s^2 + 152.4s + 115.7} \tag{4.38}$$

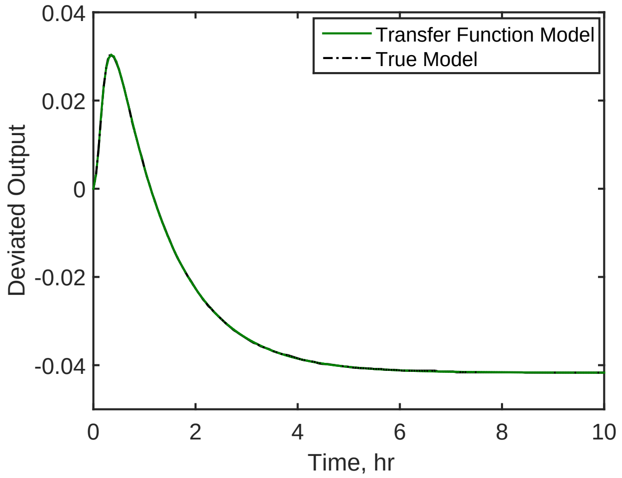


Figure 4.21: Model fitting

The transfer function of second order characteristic equation yielded a percent fit of 97.85% and mean square error (MSE) of 1.692×10^{-07}. But a transfer function with third order characteristic equation gave a percent fit of 99.47% and MSE of 1.012×10^{-08}. The third order fit between deviated output of transfer function and time is shown in Fig.4.21. The figure also shows the inverse response in the output of the transfer function. The cause of the inverse response is the presence of positive zeroes, which are 0.945 and 311.05.

The third order process was converted to a combination of a first order and second order process.

$$y(s) = G_c(s)\left(\frac{-0.5621(9.74 \times 10^{-03}s + 1)}{7.7 \times 10^{-03}s^2 + 0.197s + 1} + \frac{0.979}{1.1205s + 1}\right)\bar{y}_{sp}(s) \tag{4.39}$$

where K_1 and K_2 are -0.5621 and -0.979, τ,τ_1 and τ_2 are 9.74×10^{-03}, 0.088 and 1.1205 respectively, and ζ is 1.12. The transfer function of the compensator for the process will be

$$y'(s) = G_c(s)k\left(\frac{1}{1.12057s+1} - \frac{9.74 \times 10^{-03}s+1}{7.7 \times 10^{-03}s^2+0.197s+1}\right)\bar{y}_{sp}(s) \tag{4.40}$$

The value of k can be estimated using the above proposed method. In this case, as $K_1 > K_2$, $\tau_1^2 < \tau\tau_2$ and $\tau+\tau_2 > 2\zeta\tau$, so

$$k < \frac{K_2\tau_1^2 - K_1\tau\tau_2}{\tau_1^2 - \tau\tau_2} < 0.442 \tag{4.41}$$

$$k < \frac{K_1(\tau+\tau_2) - 2K_2\tau_1\zeta}{\tau+\tau_2-2\tau_1\zeta} < -0.474 \tag{4.42}$$

The common solution for k from Eq: 4.41 and 4.42 was found to be $k < -0.474$.
The case study has been extended to the process having the problem of inverse response and a dead time. To study this it is assumed that the process is having a dead time of 0.1 hr.

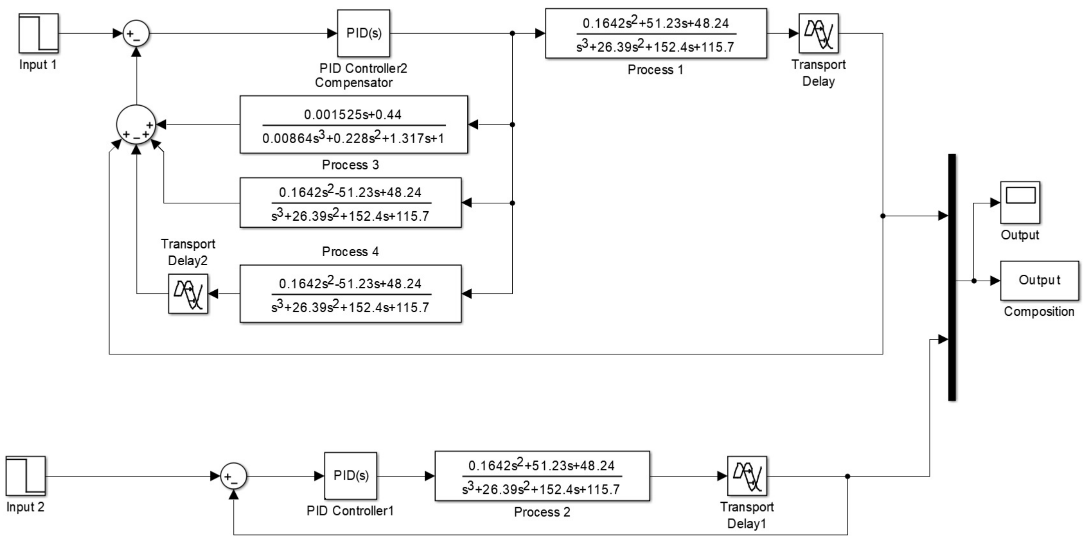


Figure 4.22: SIMULINK® Model of Closed Loop having Compensator

4.2.3 Closed Loop Results

The feedback loop for controlling the product conversion was developed using PI controller. The controller was tuned using Ziegler-Nichols method and the estimated controller parameters are $P = 1.268$ and $I = 1.725$. To eliminate the inverse response the compensator gain was considered as -0.475. The overall transfer function of compensator is shown as.

$$y'(s) = \frac{1.525^{-03}s^2 + 0.44s}{8.64 \times 10^{-03}s^3 + 0.228s^2 + 1.317s + 1} \tag{4.43}$$

while for the compensation of both inverse response and dead time problems, the overall transfer function of compensator can be represented as.

$$y'(s) = (1 - e^{-0.1s})\left(\frac{0.1642s^2 - 51.23s + 48.24}{s^3 + 26.39s^2 + 152.4s + 115.7}\right) + \left(\frac{1.525^{-03}s^2 + 0.44s}{8.64 \times 10^{-03}s^3 + 0.228s^2 + 1.317s + 1}\right) \tag{4.44}$$

The closed loop model of the process with and without using compensator was developed in SIMULINK® software as shown in Fig. 4.22. The closed loop is studied by giving step changes in set point of the process. The process is also analyzed when various model mismatch conditions are given to the process.

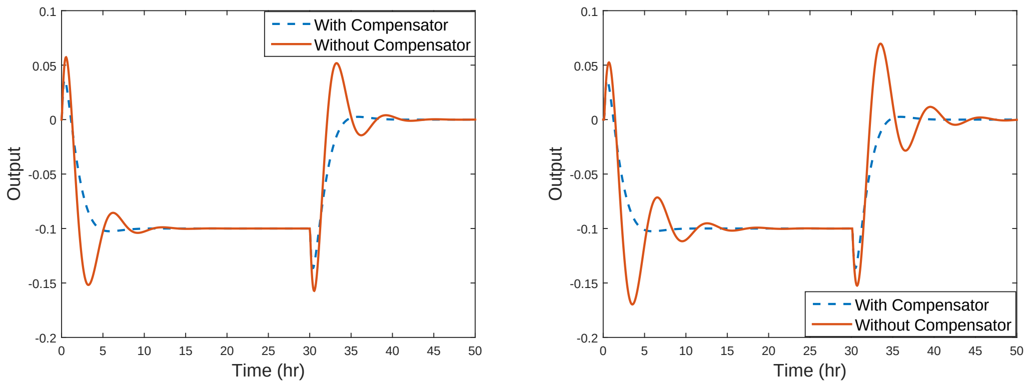


Figure 4.23: Closed loop response for set point change by $\pm 10\%$ (a) Only inverse response (b) Both inverse response and dead time

4.2.3.1 Servo Response

The servo response was studied by giving a step change in the set point of $\pm 10\%$ for both the cases i.e. process with and without compensator. It can be seen in Fig. 4.23 that process with compensator gives better results with less overshoot and less oscillations for both the cases of inverse response and dead time. The integral errors for the closed response is shown in Table: 4.8, it can be observed that errors are decreasing on using the compensator.

Table 4.8: Integral errors for $\pm 10\%$ step change

	Inverse Response		Inverse Response & Dead Time	
	With Compensator	Without Compensator	With Compensator	Without Compensator
ISE	0.046	0.060	0.049	0.073
IAE	0.507	0.675	0.537	0.904
ITAE	8.130	11.470	8.784	16.457

4.2.3.2 Change in Compensator Gain

The compensator gain which was decided to be as -0.475 is given $\pm 20\%$ change for checking the robustness of both the compensators. Fig. 4.24 shows the change in closed loop response when gain is changed from -0.475 to -0.38 and -0.57. When k value is changed to -0.57 then according to Eq. 4.42 the system is still showing an inverse response. This is the reason of getting some sluggish response than other two values. It can also be observed from the integral errors which are shown in Table. 4.9 that the error values are higher for both the compensators when k is -0.57 rather than other two gains. Since despite of the changes given in the compensator gains, the process is getting controlled after certain time and errors are also not varying to the high extents; this makes the compensator to be robust during changes in compensator gain.

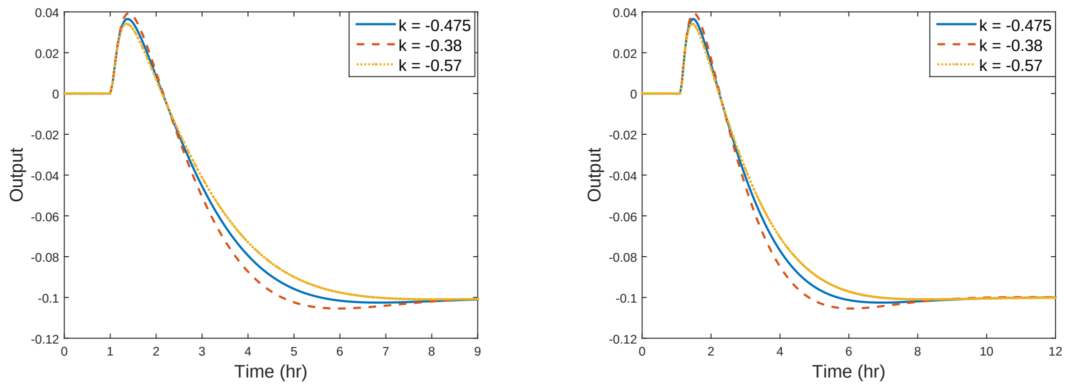


Figure 4.24: Change in compensator gain by $\pm 20\%$ (a) Only inverse response (b) Both inverse response and dead time

Table 4.9: Integral errors for $\pm 20\%$ change in compensator gain

	Inverse Response			Inverse Response & Dead Time		
	k = -0.475	k = -0.380	k = -0.570	k = -0.475	k = -0.380	k = -0.570
ISE	0.024	0.024	0.024	0.025	0.025	0.025
IAE	0.260	0.253	0.272	0.270	0.263	0.282
ITAE	0.354	0.340	0.392	0.381	0.366	0.419

4.2.3.3 Process Model Mismatch

The robustness of the compensator and controller was also studied by considering the model mismatch situation. The changes in processes gains, K_1 and K_2, and the time constants,τ_1 and τ_2, of both the processes (1^{st} and 2^{nd} order processes) were considered for the study.

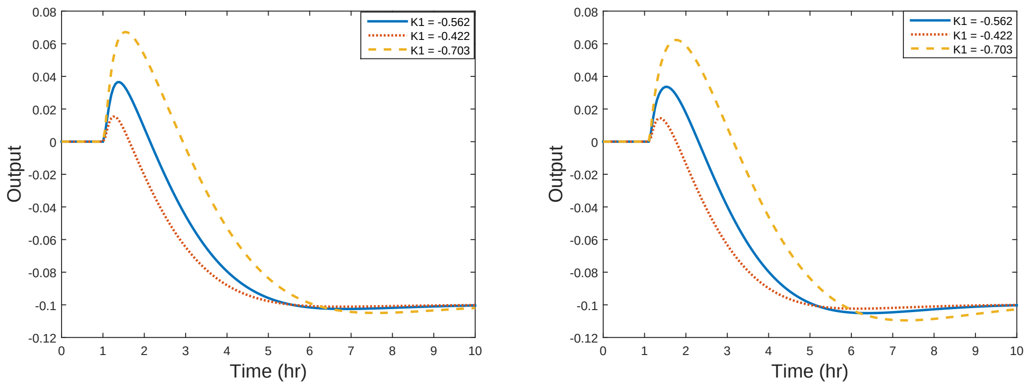


Figure 4.25: Uncertainty in Process Gain K_1 by $\pm 25\%$ (a) Only inverse response (b) Both inverse response and dead time

4.2.3.4 Uncertainty in Process Transfer Function Gain

The model mismatch was studied by giving change in both the process gains by $\pm 25\%$. The mismatch was studied by giving negative step change in set point for both the closed loops having just inverse response compensator and loop with inverse response plus dead time compensator. It can be seen in Fig. 4.25 that the process is getting controlled in both the positive and negative changes in K_1 with some variations from the normal gain value. In the case of K_2, as shown in the Fig. 4.26, the negative change is bringing high overshoot and sluggishness in the response. However every time process is getting

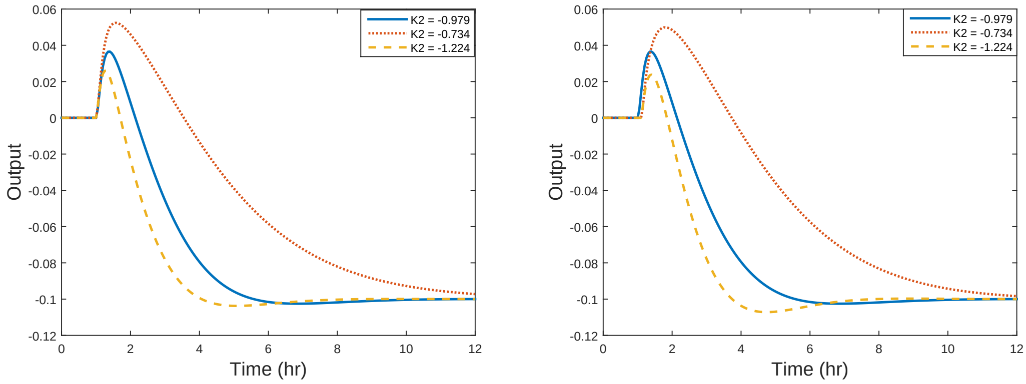


Figure 4.26: Uncertainty in Process Gain K_2 by $\pm 25\%$ (a) Only inverse response (b) Both inverse response and dead time

controlled making the process robust to the changes in process gain. Integral errors for the process model mismatch are given in Table. 4.10. The errors values are high in the case of higher K_1 values and lower K_2 values.

Table 4.10: Integral errors for uncertainty in Process Gains of the processes

	Inverse Response			Inverse Response and Dead Time		
	K_1 = -0.562	K_1 = -0.422	K_1 = -0.703	K_1 = -0.562	K_1 = -0.422	K_1 = -0.703
ISE	0.023	0.014	0.045	0.025	0.015	0.048
ITE	0.254	0.185	0.395	0.268	0.191	0.421
ITAE	0.338	0.215	0.649	0.384	0.227	0.779
	K_2 = -0.979	K_2 = -0.734	K_2 = -1.224	K_2 = -0.979	K_2 = -0.734	K_2 = -1.224
ISE	0.023	0.058	0.014	0.025	0.059	0.015
ITE	0.254	0.584	0.167	0.268	0.588	0.179
ITAE	0.338	1.583	0.168	0.384	1.573	0.201

4.2.3.5 Uncertainty in Process Transfer Function Time Constants

As model is carrying two processes simultaneously, the model robustness is verified by changing time constants of both the processes by $\pm 25\%$. The response during change in process time constants for both the cases are shown in Fig. 4.27 and 4.28. In both the cases, response is showing controlled results with variable overshoot and settling time. The integral errors for the responses are shown in Table. 4.11. It can be observed from the integral errors that the deviation in the errors is very low in all the cases, so the system is robust to the time constants mismatch.

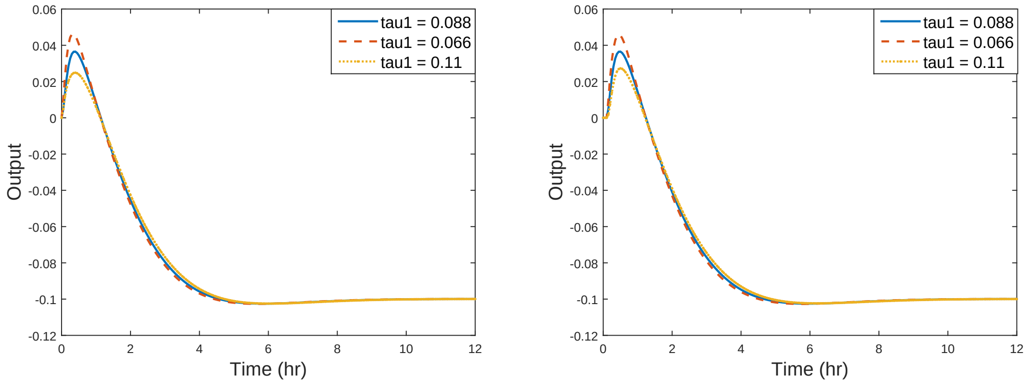


Figure 4.27: Uncertainty in τ_1 for process with (a) Inverse response (b) Inverse response and dead time

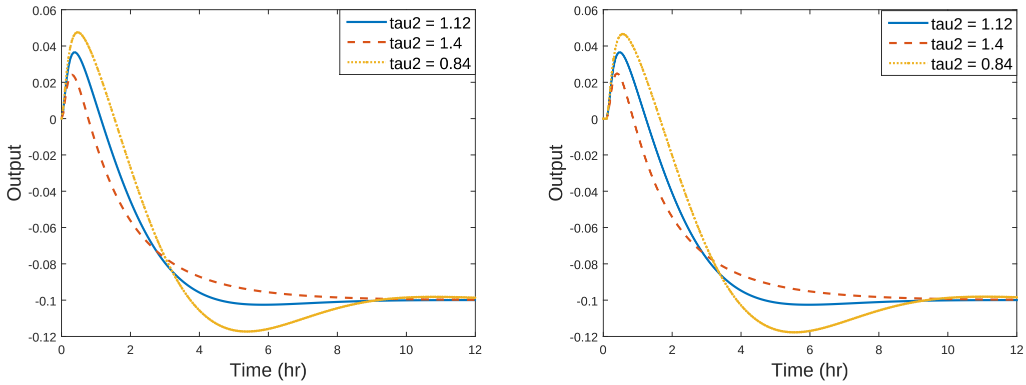


Figure 4.28: Uncertainty in τ_2 for process with (a) Inverse response (b) Inverse response and dead time

4.3 Conclusions

In this chapter, the problem of inverse response due to step change in load variable and manipulated variable in the RDC was considered. In the case of inverse response due to the load variable, the situation was handled using Feedforward-Feedback controller. The feedforward controllers were developed by estimating the transfer functions of the process and both the load disturbances. When the process was considered in the form of a transfer function model, the FFFB controllers gave excellent results with small integral errors compared to just FB controller. But when the same controllers were linked to control the S-function model of the plant, the controllers gave mixed results. These mixed results lead to restrict the use of FFFB controllers in the case of inverse response

Table 4.11: Integral errors for uncertainty in time constants of the processes

	Inverse Response			Inverse Response and Dead Time		
	τ_1 = 0.088	τ_1 = 0.11	τ_1 = 0.066	τ_1 = 0.088	τ_1 = 0.11	τ_1 = 0.066
ISE	0.023	0.022	0.024	0.025	0.023	0.026
ITE	0.260	0.259	0.260	0.270	0.269	0.270
ITAE	0.354	0.371	0.341	0.381	0.395	0.368
	τ_2 = 1.120	τ_2 = 1.4	τ_2 = .0.84	τ_2 = 1.120	τ_2 = 1.4	τ_2 = .0.84
ISE	0.023	0.033	0.018	0.025	0.034	0.019
ITE	0.260	0.350	0.245	0.270	0.364	0.255
ITAE	0.354	0.711	0.417	0.381	0.771	0.447

in the load variable. In the case of inverse response due to the step change in manipulating variable, a case study of RDC was taken from Peng et al. (2003). A compensator was designed for the 3^{rd} order transfer function model that addressed both the problems of inverse response and dead time in the process. The proposed methodology of designing a compensator gave better results with small integral errors. The robustness of process and compensator was also studied by giving model mismatch situations with the maximum deviation of $\pm 25\%$ in the closed loop. In all the cases the process was controlled with small deviations from the true characteristics.

Chapter 5

Development of Soft Sensor

The real time estimation of process efficiency is a preferable condition for the advanced process control techniques. In the case of distillation, its efficiency can be signified by the amount of prime component present in the product streams. For the estimation of product composition, a hardware composition sensors such as Gas Chromatograph (GC) can be used. Since GC or other hardware sensors carries time lag in their process, they are not preferred for the online estimation work. On the other hand, tray temperatures can easily be estimated using hardware sensors such as thermocouples. The tray temperatures are also the prime factor which affects the product composition in the column. These tray temperatures can relate to the product estimation if they are regressed correctly. In soft sensing, these tray temperatures are regressed with various techniques like PCA, PLS, ANN etc. for the estimation of product composition. As RDC column is a highly nonlinear dynamic unit, the soft sensor also needs to be a dynamic and nonlinear in nature to cover RDC at all the working conditions.

Generally, distillation columns are controlled by controlling the first or last tray temperature of the column. For getting tighter control on the RDC, the column needs to have direct control on composition instead of indirect control using tray temperatures. The direct control of butyl acetate composition in the product stream of the RDC is proposed in the study with the online estimation from the soft sensor.

This chapter starts with the introduction to recurrent neural network (RNN) technique. The RNN technique has been used to estimate the butyl acetate composition in the bottoms product stream of RDC. The RNN based sensor is tested for the step changes for both the open and closed loop of the plant. The study has also been extended to the use of Pseudo Random Binary Sequence (PRBS) signal for checking the soft sensor efficiency at practical situations.

5.1 Recurrent Neural Network(RNN)

A recurrent neural network is a type of dynamic neural network which takes temporal input to produce temporal outputs. The most substantial feature of the RNN is that the current output of the network not only depends upon the current input of the network but also depends upon the output that was produced in previous time interval. This makes the network adaptive to some extent. This feature make it unique from the other dynamic networks.

In RNN, the output of the network at any time, t, is generated from the network input

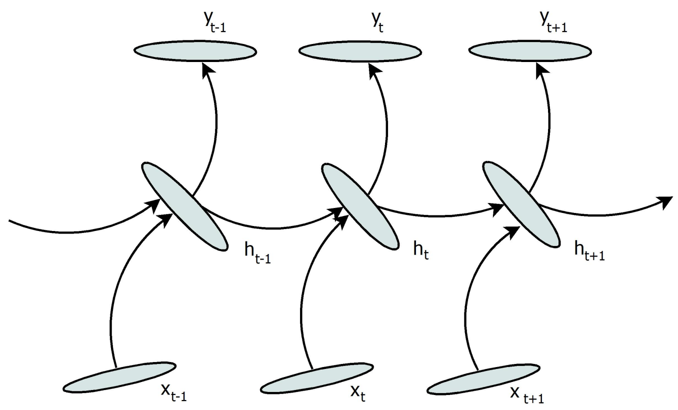


Figure 5.1: RNN unfolded in time

data at time, t, and the hidden weight at time, $t-1$. When a network has a x_t input, y_t output and h_t hidden state weight where subscript, t, denotes the time, then RNN can be represented as (Pascanu et al., 2013):

$$h_t = g_h(x_t, h_{t-1}) \tag{5.1}$$

$$y_t = g_o(h_t) \tag{5.2}$$

where g_h is a hidden layer transition function and g_h is an output function. θ_h and θ_o are the set of parameters in both the functions.

When a set of N training samples are given, $P = \{((x_1^{(1)}, y_1^{(1)}), (x_2^{(2)}, y_2^{(2)}), ..., (x_{T_n}^{(n)}, y_{T_n}^{(n)}))\}_{n=1}^{N}$, then parameters of the network can be estimated by minimizing the following cost function:

$$J(\theta) = \frac{1}{N} \sum_{n=1}^{N} \sum_{n=1}^{T_n} d(y_t^{(n)}, f_o(h_t^{(n)}) \tag{5.3}$$

where $h_t^{(n)} = g_h(x_t^{(n)}, h_{t-1}^{(n)})$ and $h_0^{(n)} = 0$. $d(a, b)$ is a predefined divergence measure between a and b, like cross entropy or Euclidean distance.

When an RNN is unfolded over the time as shown in the Fig. 5.1, the network becomes deep as the input has to pass through the various non-linear hidden computational paths to get the adequate output. When hidden layer transition function and output functions are expanded then RNN model can be illustrated as:

$$h_t = g_h(x_t, h_{t-1}) = \phi_h(H^\top h_{t-1} + X^\top x_t) \tag{5.4}$$

$$y_t = g_o(h_t) = \phi_o(Y^\top h_t) \tag{5.5}$$

where X and Y are the input and output matrices and H is a transition matrix. ϕ_h and ϕ_o are the element wise hidden and output layer nonlinear functions. Generally saturating nonlinear functions are used like hyperbolic tangent function and logistic sigmoid function for element wise hidden transition function.

Table 5.1: Column Specifications for Soft Sensor Studies

Parameters	Values
Feed Flow Rates	
Acetic Acid	10.5 mol/h
n-Butanol	9.5 mol/h
Presure	1 atm
Number of Stages	15
Reactive Zone	Tray number 6-9 [1]
Feed Stage Location	
Acetic Acid	Tray number 6
n-Butanol	Tray number 9
Reboiler Duty	1.67 kW

5.1.1 RNN Based Soft Sensor

The dynamic model of RDC was developed in MATLAB®/SIMULINK® for the soft sensor study. Firstly the dynamic plant was initialized from the steady state results of

[1] Tray numbered from top to bottom

the model. Then the dynamic RDC model was simulated for the collection of temporal tray temperatures and *n*-Butyl Acetate bottoms product composition data. The column specifications considered for the modeling purpose are shown in Table 5.1.

The sequential data of tray temperatures and n-Butyl Acetate bottoms product compo-

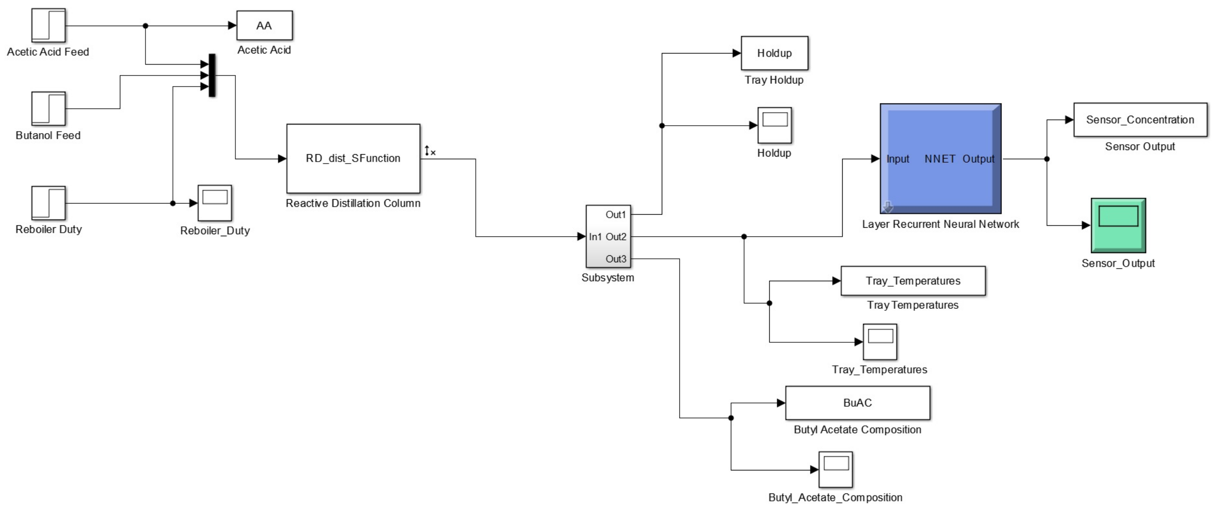


Figure 5.2: Open loop SIMULINK® diagram of RDC with soft sensor

sition was obtained from the open loop model of the RDC at various conditions. This data was gathered and used for training the network. The data obtained contained 99 sets of model run, each containing 15 tray temperatures and 1 product composition at the sample time of 15 minutes for 12 hour long run of the model. The data is collected by varying acetic acid, butanol feed composition in the range of 9.5-11.5 mole/hr and 8.5-10.5 mole/hr and varying the reboiler duty from 0.27-2.77 kW. The data was divided into 56/22/21 parts for training, validating and testing the network, respectively.

The RNN used for the sensing purpose consisted of an input layer with 15 tray temperature variables, single hidden layer with 5 numbers of neurons and an output layer with a single variable showing butyl acetate bottoms composition. The number of neurons was optimized by minimizing the mean square error (MSE) of the data. Levenberg-Marquardt algorithm was used for training the network. After training the network, it was observed that the training MSE came to be a small value of 7.16×10^{-11}. The R^2 value of 0.999 was obtained between actual composition and network output for the testing as well as validation of the network, thus making this network to be appropriate for the sensing work. Dynamic behaviour of the network was also validated by comparing open loop and closed loop responses of the model with the soft sensor results.

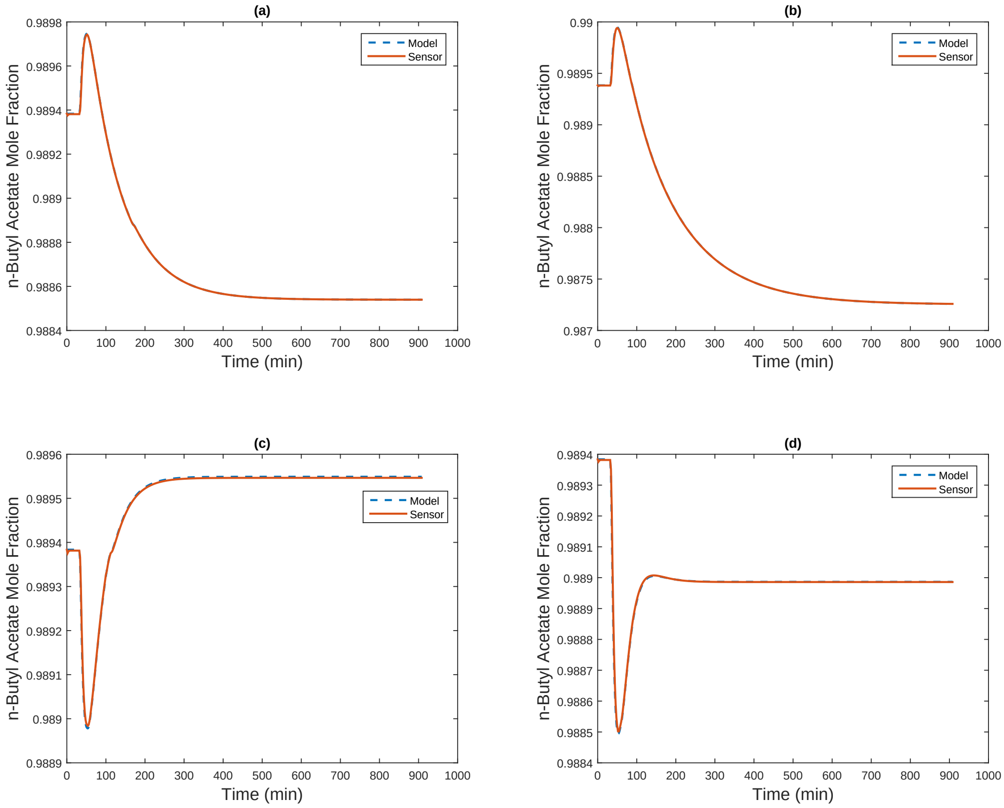


Figure 5.3: Open Loop Response for Step Change in Acetic Acid Feed Flow Rate by $(a) - 5\%$ $(b) - 8\%$ $(c)5\%$ $(d)10\%$

5.1.2 Open Loop Response of the Model

The open loop response of the model was studied by introducing the step disturbances in the input flow rate of reactants. The SIMULINK® open loop model of the RDC is shown in Fig. 5.2. The blue block in the model is a RNN based soft sensor which is getting inputs as tray temperatures from the subsystem and producing butyl acetate composition as a sensor output. The soft sensor performance is judged on the basis of its prediction accuracy when compared to the true composition of the model. The estimation accuracy is quantified on the basis of mean square error (MSE) between soft sensor output and true model composition value of butyl acetate in the bottoms stream of the RDC.

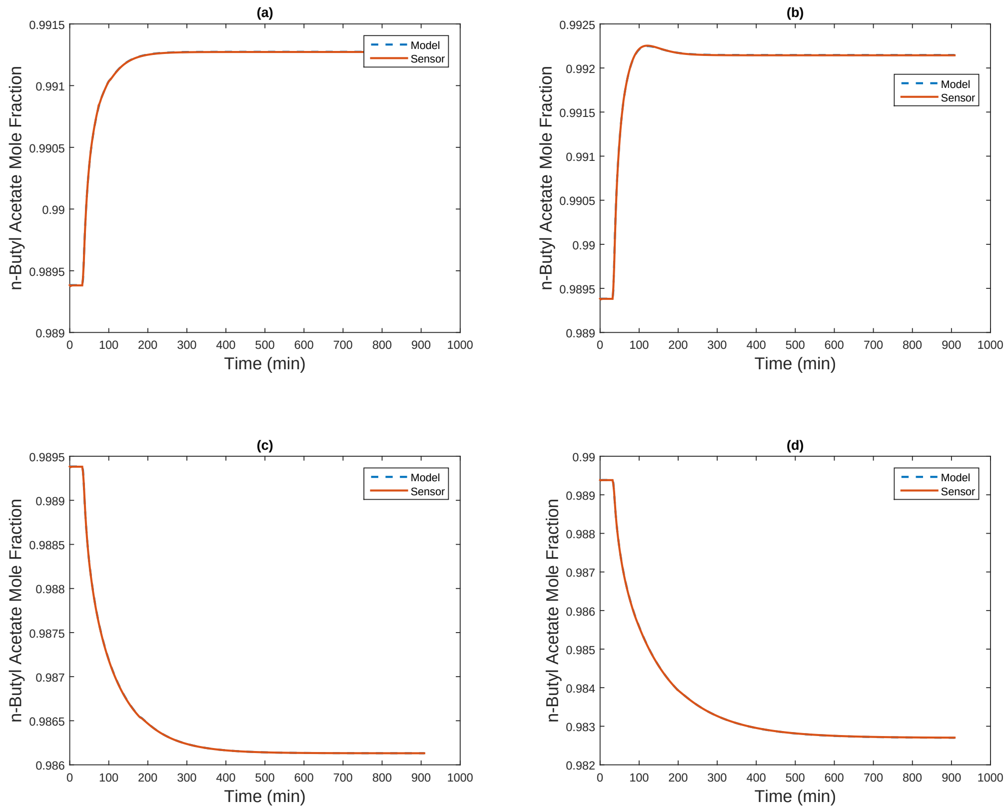


Figure 5.4: Open Loop Response for Step Change in Butanol Feed Flow Rate by $(a)-5\%$ $(b)-10\%$ $(c)5\%$ $(d)8\%$

5.1.2.1 Step Change in Acetic Acid Feed Flow Rate

During the steady state run, the base value of Acetic Acid feed flow rate was fixed to be 10.5 mole/h with n-Butyl Acetate bottoms mole fraction of 0.98938. The effect on bottom product composition was studied by adding step disturbances in the feed flow rate of Acetic Acid as shown in Fig. 3.6. The composition of *n*-Butyl Acetate is getting affected at different disturbances in the feed flow rate. It can also be observed from Fig. 3.6 (a), (b) and (c) that the open loop response for the system is showing inverse response indicating the model to be sensitive for the large disturbances. The inverse response in the system is because of two opposing processes working simultaneously. The first process is that on increasing the acetic acid quantity, the excess acetic acid reacts with remaining butanol leading to increase the butyl acetate composition and secondly increase in acetic acid increases the heavier compound at bottom of the column leading to the decrease in product purity. Similarly the phenomenon can be applied to

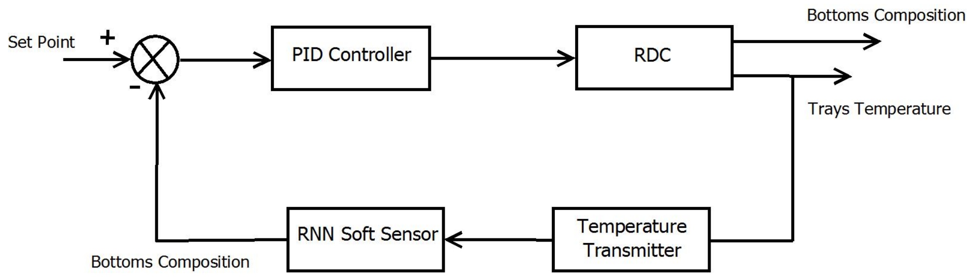


Figure 5.5: Closed loop

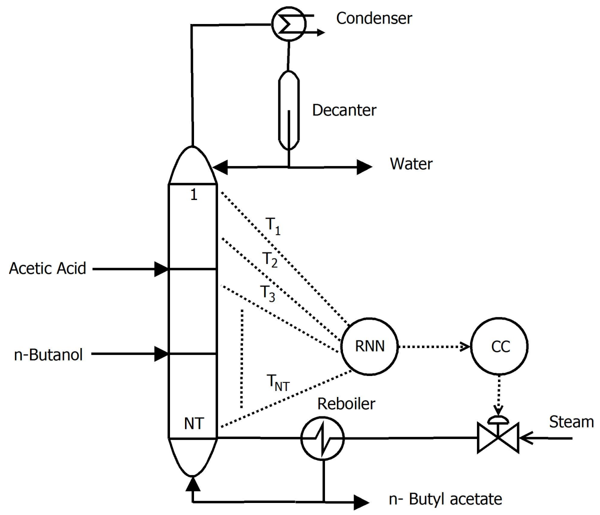


Figure 5.6: Control structure of RDC

Fig. 3.6 (b) and (c). It can be noticed that soft sensor predicted composition and the model composition are almost overlapping each other. The MSE is computed to be 6.58×10^{-12} for -5%, 1.62×10^{-11} for -8%, 1.31×10^{-11} for +5% and 3.51×10^{-11} for +10% step change.

5.1.2.2 Step Change in Butanol Feed Flow Rate

The steady state value of n-Butanol feed flow rate is 9.5 mol/h for n-Butyl Acetate mole fraction of 0.98938 in the bottoms product. When step disturbance is added to the feed flow rate of n-Butanol, the variations can be observed in the n-Butyl Acetate bottom product composition as shown in Fig. 3.7. The open loop response also shows that soft sensor estimation and model output are in well agreement with the MSE of 5.52×10^{-12} for -5 %, 1.90×10^{-11} for -10 %, 5.55×10^{-12} for +5 % and 1.99×10^{-11} for +8% step change. It is observed from the MSE pattern that the error gets increased by increase in the disturbance.

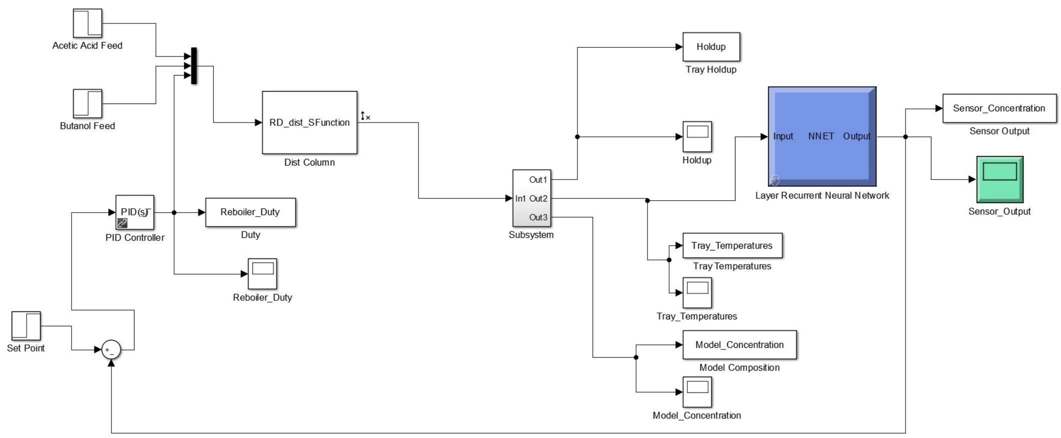


Figure 5.7: Closed loop SIMULINK® diagram of RDC with soft sensor

5.1.3 Closed Loop Response of the Model

It has been observed in the previous section that step disturbances in the load variables deviates the product composition from its steady state value. To control the n-Butyl Acetate mole fraction in the bottoms product, a PI controller with the proportional gain of 4.61 and integral time of 177.45 hr has been purposed. The composition is controlled by manipulating the reboiler duty of the column. The controller was tuned using automatic tuning toolbox of MATLAB® which tune the PI gains by choosing the crossover frequency according to the plant dynamics and designs for the phase margin with the target value of $60°$. The control structure of RDC is shown in Fig. 5.6 In the closed loop, soft sensor acts as a measuring element for the direct control of the bottoms composition as shown in Fig. 5.5. The SIMULINK® closed loop model of the RDC is shown in Fig. 5.7. It can be noticed from the SIMULINK® model that the sensor output which is a product composition is going to the comparator for the calculation error between set point value and process output value. Further servo and regulatory responses are studied to check the performance of a controller and a soft sensor that how adequately the soft senor is acting as a measuring element in the loop.

5.1.3.1 Load Change in Acetic Acid Feed Flow Fate

The load changes were given in the Acetic Acid feed flow rate by $\pm 5\%$, -8% and $+10\%$ at 30 min. The closed response for the negative load change is shown in Fig. 5.8 and that for positive load change is shown in Fig. 5.9. The response is sluggish but the

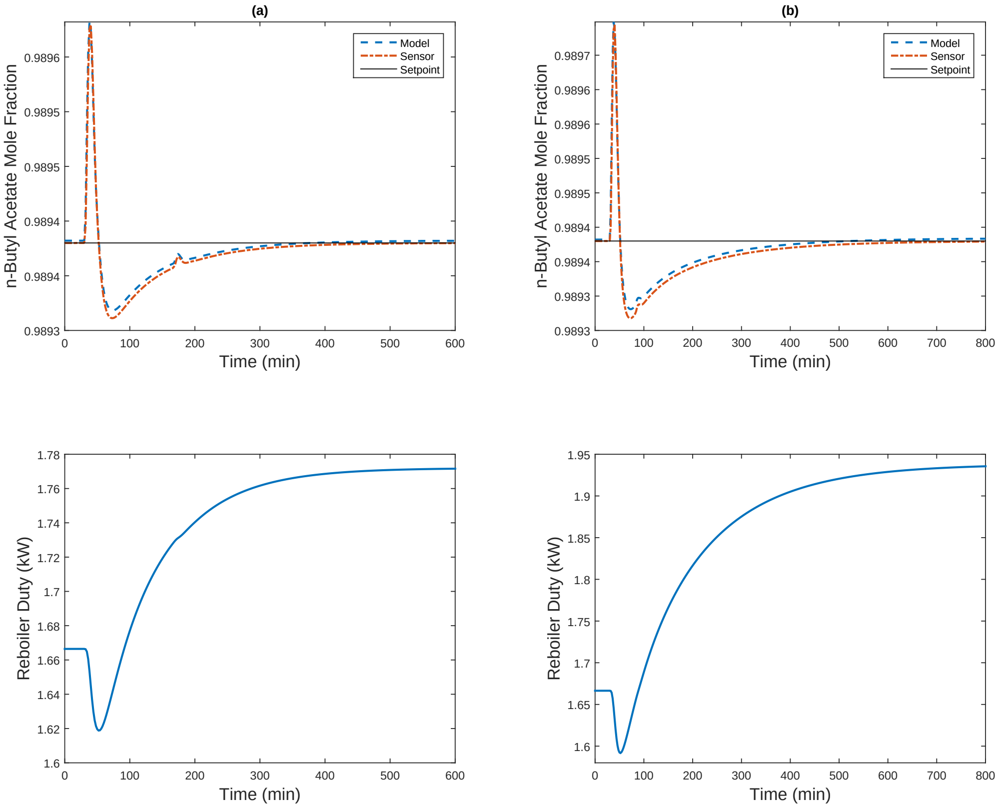


Figure 5.8: Closed loop response for negative load change in Acetic Acid flow rate by (a) -5% (b) -8%

composition is getting controlled within the time span of 600 min. It can be observed that soft sensor readings and model readings are similar to each other. The MSE between the readings are 1.18×10^{-11} for -5%, 4.02×10^{-11} for -8%, 1.26×10^{-11} for +5% and 2.52×10^{-11} for +10% step change.

5.1.3.2 Load Change in Butanol Feed Flow Rate

To study the closed response of the system in the presence of step disturbances in *n*-Butanol feed flow rate, step changes of $\pm 5\%$, -10% and $+8\%$ were given to the initial rate. The closed loop response during the negative and positive step changes in the butanol composition are shown in Fig. 5.10 and 5.11, respectively. In both the cases, bottoms composition is brought to the set point within 200 min. Soft sensor estimation also shows a good agreement with the model values with the small MSE values of 3.38×10^{-11} for -5%, 3.70×10^{-10} for -10%, 8.90×10^{-11} for 5% and 2.32×10^{-10} for 8% step change. It can also be observed that the value of error increases as the

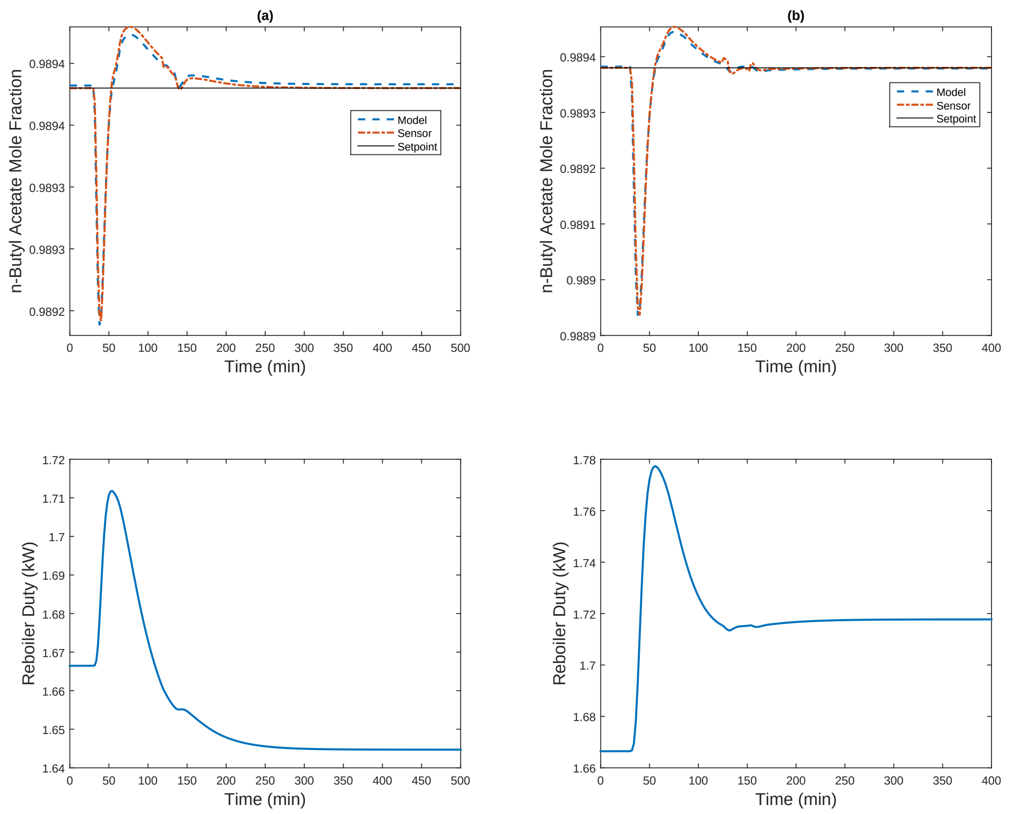


Figure 5.9: Closed loop response for positive load change in Acetic Acid flow rate by (a) 5% (b) 10%

disturbance increases but the error is sufficiently small and can be ignored.

5.1.3.3 Set Point Change

The closed loop response was also studied for the set point change by giving a step change from 0.98938 to 0.89 (-10%) and from 0.89 to 0.98938 (10%). The closed loop response is shown in Fig. 5.12. The bottom composition is getting controlled in both the cases of positive and negative set point changes. The flat portion in the figures of manipulated variables indicates the lower and upper limit of the reboiler duty that has been set in the controller. It is observed that soft sensor works efficiently to match the closed loop servo response; the MSE is computed to be 8.46×10^{-6} for -10 % and 3.57×10^{-7} for +10 % step change.

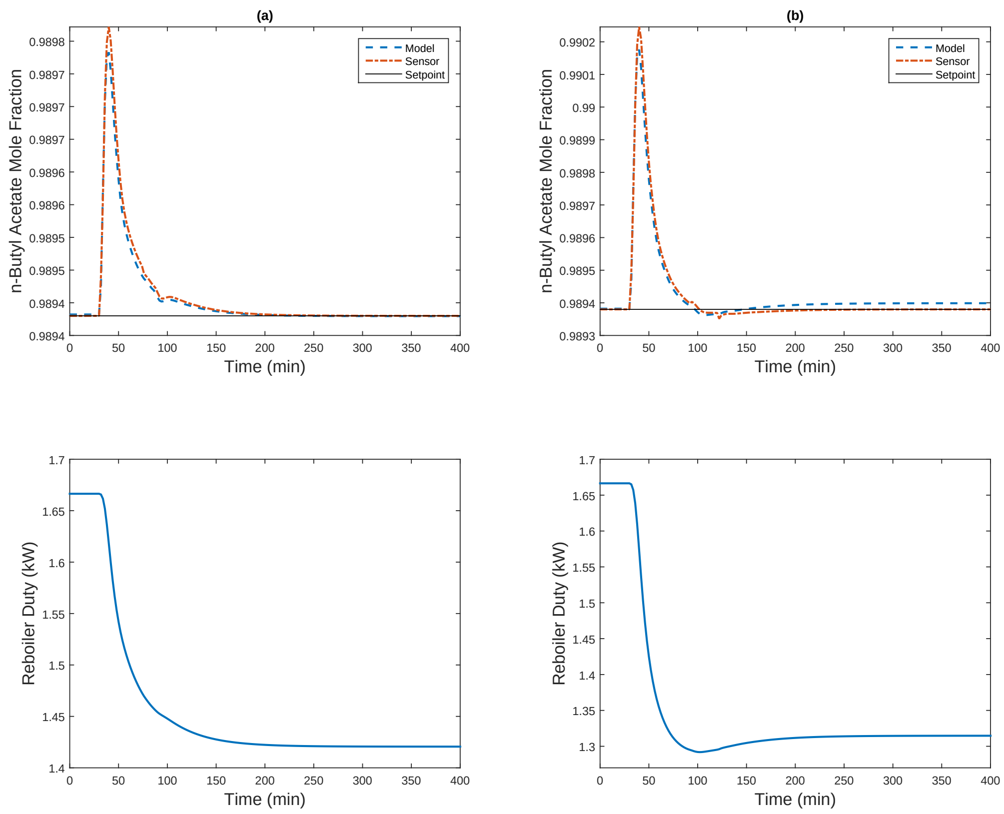


Figure 5.10: Closed loop response during negative load change in *n*-Butanol flow rate by (a) -5% (b) -10%

5.2 Performance of the Control System

The performance of the control system is judged by its robustness to the variation in the set point as well as the load change. The performance criteria was chosen to be Integral of the square of the error (ISE), Integral of the absolute value of error (IAE) and Integral of time-weighted absolute error (ITAE). All the integral errors between product output and set point are estimated using Eq. 4.5, 4.6 and 4.7

The performance of the PI controller of the control loop system has been compared using the integral errors. The integral error values for the regulatory response are shown in Table 5.2 and those for the servo response are shown in Table 5.3. It can be observed that in every scenario the error increases as the value of disturbance increases.

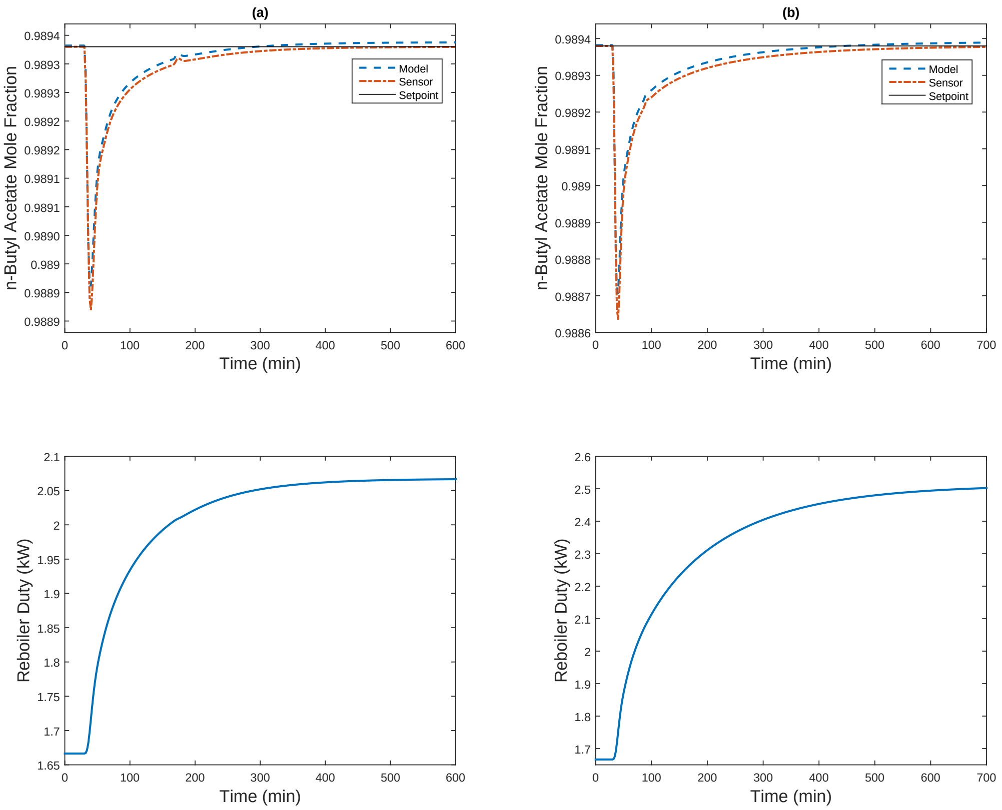


Figure 5.11: Closed loop response for negative load change in *n*-Butanol flow rate by (a) 5% (b) 8%

5.3 Pseudo Random Binary Sequence (PRBS)

In a chemical plant, it is not favorable to move away the plant from its steady state conditions by giving a step change in its load or manipulated variables. The process engineers recommended to go with impulse signal or multiple impulses in the form of pseudo random binary sequence (PRBS) (Perry and Green, 2008).

The PRBS signal is a binary signal which has a sequence length of N and a switching level of +*a* and -*a*. The minimum switching time between the levels is T_{SW} and the switching time has to be the multiple of T_{SW} (Häggblom, 2016). The general equation of PRBS is

$$N = 2^{n_r} - 1 \tag{5.6}$$

where n_r is number of shift registers. When the sample time of T_S is taken for the system than T_{SW} can be taken as

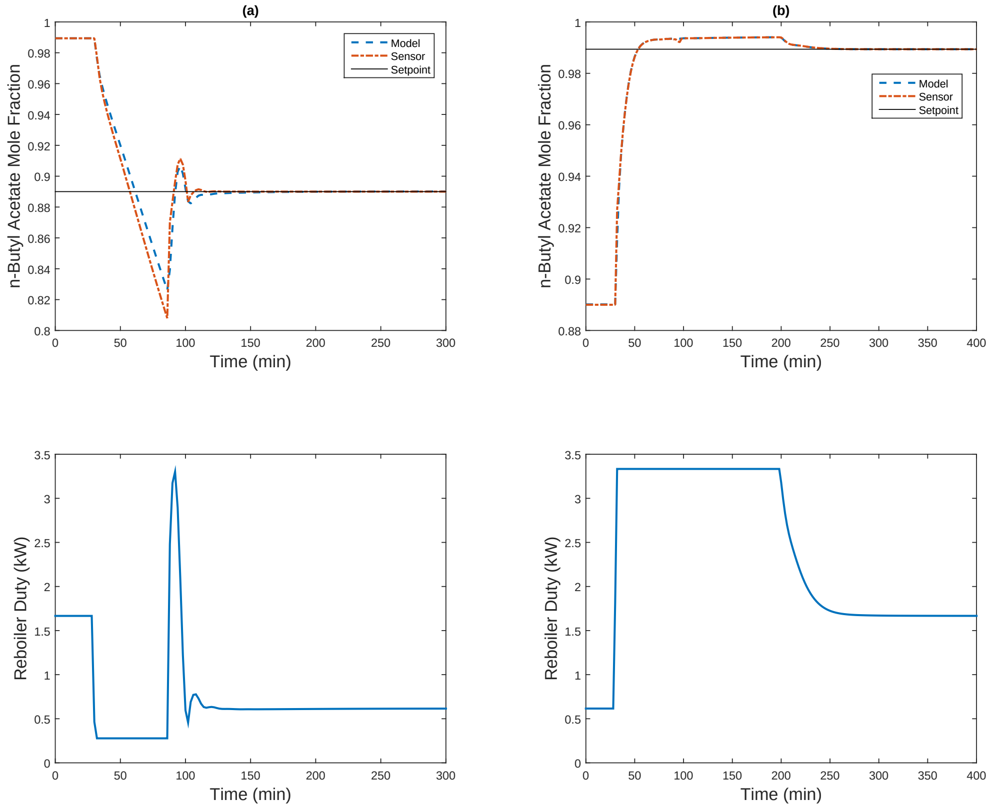


Figure 5.12: Closed loop response for set point change by (a) -10% (b) 10 %

$$T_s \approx 0.25\, T_{SW} \tag{5.7}$$

For the current study, the sample time is taken to be of 2 min, the switching time is estimated as 8 min with a sequence length of 90 and number of shift registers are calculated as 7.

5.3.1 Open Loop with PRBS Disturbance

After obtaining the perfect performance of soft sensor during the step changes in the load and manipulating variable, the soft sensor has also been tested for the disturbances in the form of PRBS signal. The PRBS signal with a disturbance magnitude of $\pm 10\%$ of the steady state value of acetic acid and butanol feed rate and reboiler duty is used as shown in the Fig. 5.13 (d). The plant responded to the PRBS disturbance with the variation in the response of product composition. When the response between soft sensor and true model value is compared, it can be observed from Fig. 5.13 (a), (b) & (c) that soft sensor estimations gave accurate results with small deviation from true values.

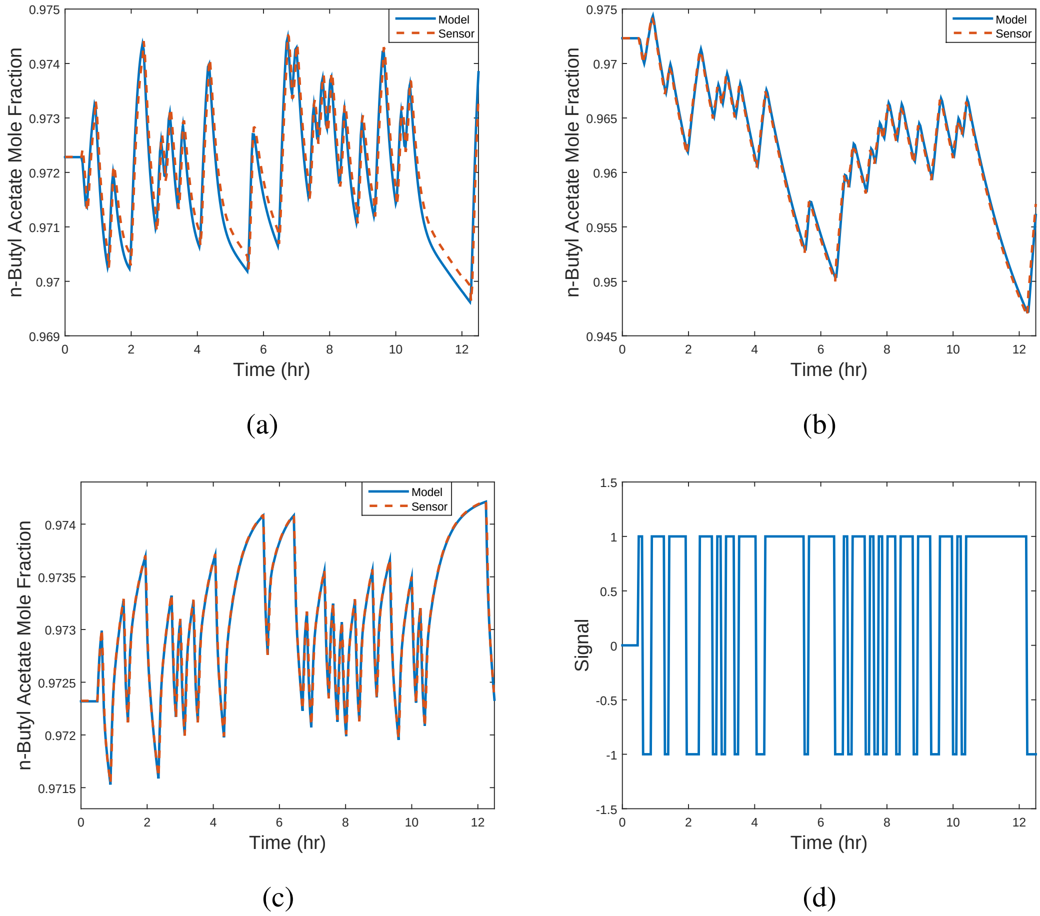


Figure 5.13: Model and sensor comparison during (a) Load change in Acetic Acid feed rate (b) Load change in Butanol feed rate (c) Change in reboiler duty (d) PRBS signal

The mean percentage error for the change in acetic acid , butanol feed rate and reboiler duty is -0.01%, -0.001% and 0.009%, respectively. These small values suggest that soft sensor is a reliable model which can be used as measuring element in the closed loop to control the product composition directly.

5.4 Conclusions

In this chapter, a soft sensor for an RDC has been developed to estimate the butyl acetate composition in the bottoms stream using the tray temperatures of the column. The dynamic recurrent neural network (RNN) technique was applied for the estimation. The sensor was designed and tested for the open loop step changes and PRBS disturbance signals. The sensor worked perfectly for the open loop changes in the column and it was further tested for the closed loop response. The closed loop was developed for

Table 5.2: Performance criteria for load change in Acetic Acid and Butanol feed flow rate

Acetic Acid	ISE	IAE	ITAE	Butanol	ISE	IAE	ITAE
5%	3.52×10^{-06}	0.0274	5.1649	5%	6.40×10^{-07}	0.0159	6.007
8%	9.13×10^{-06}	0.0435	6.8948	10%	2.32×10^{-06}	0.0224	6.9554
-5%	2.08×10^{-06}	0.0218	6.6641	-5%	9.96×10^{-07}	0.0227	6.6144
-10%	7.21×10^{-06}	0.0174	6.8252	-8%	2.21×10^{-06}	0.0307	7.6215

Table 5.3: Performance criteria for set point change

	ISE	IAE	ITAE
-10%	0.1078	2.4518	119.1762
10%	0.0220	1.1543	76.300

directly controlling the product composition using soft sensor as a measuring element in the loop. It was observed that the sensor gave close to perfect results with the true composition when sensor is linked with PI for the control purpose. In both the cases of open and closed loop, the sensor results are proved good on the basis of small MSE.

Chapter 6

Dynamic Neural Network based Model Predictive Control

The advance control techniques play a crucial role in the working of a process industry. Even with the use of the advanced controllers in a less quantity in a plant as compared to conventional PID controllers, they serves at higher capital as compared to conventional controllers.

In the case of our study, RDC has been showing inverse response due to step change in acetic acid feed rate (load variable). The inverse response problem can be cured with the controller having feedforward nature or with future prediction characteristics. The technique of getting a feedforward feedback controller proved to be not a reliable option in Section 4.1.7 because of the highly nonlinear nature of RDC. This chapter considers second option of future prediction by studying a model predictive control technique for the control of product composition of an RDC.

This chapter starts with the study of model predictive control (MPC) technique for controlling the RDC. The MPC needs an internal model for the future prediction purpose which can be a transfer function, state space or some other non linear model like neural network based models. The model has to be accurate as the control action depends on the future predictions from the model. So the model has to be nonlinear and dynamic in nature to cover all the range of RDC at every condition. This study involves a dynamic neural network based RDC model which has been used inside the MPC. After optimizing the MPC parameters, the MPC is used to control the butyl acetate compostion in the bottoms stream of the RDC. The closed loop MPC results are compared with PI results for both the step changes and PRBS disturbance signals. The study is also extended with the inclusion of a dead time in the system and checking the robustness of the MPC during dead time presence.

6.1 Model Predictive Control

Model predictive control is a control strategy which works on the internal process model and an optimizer as shown in Fig. 6.1. The role of estimator is to estimate the model outputs for the N_p number of future sampling intervals and pass it to the optimizer. The optimizer use these outputs and previous input data to estimate the plant input for the N_c number of future control intervals by minimizing the cost function as mentioned below:

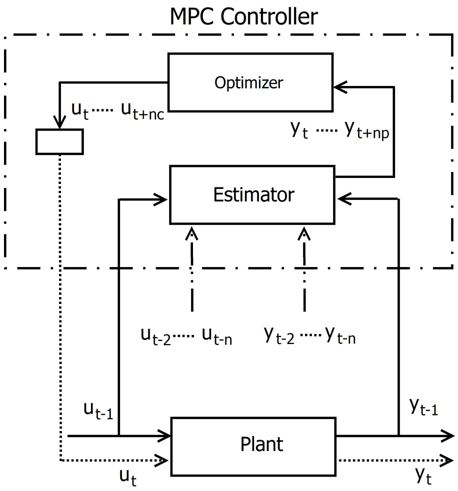


Figure 6.1: Plant with MPC

$$J(u^C) = j_y + j_{\Delta U} \tag{6.1}$$

$$j_y = \sum_{i=1}^{N_p} W_y\{y(k+i|k) - y_{ref}(k+i|k)\}^2 \tag{6.2}$$

$$j_{\Delta U} = \sum_{j=0}^{N_c} W_{\Delta U}\{U(k+i|k) - U(k+i-1|k)\}^2 \tag{6.3}$$

$$y_{min} \leq y \geq y_{max} \tag{6.4}$$

$$u_{min} \leq u \geq u_{max} \tag{6.5}$$

The cost function contains two parts, one is sum of square of deviation of output variables, *y*, from the set point values, y_{ref}, and second part is sum of square of deviation of previous and current input variable, *u*, at current control interval *k*. The future input value of the process is optimized by minimizing the sum of output deviation for N_p future predictions and change in input is minimized for N_c future steps. It is assumed that plant input becomes constant after N_c steps. After minimizing the cost function,

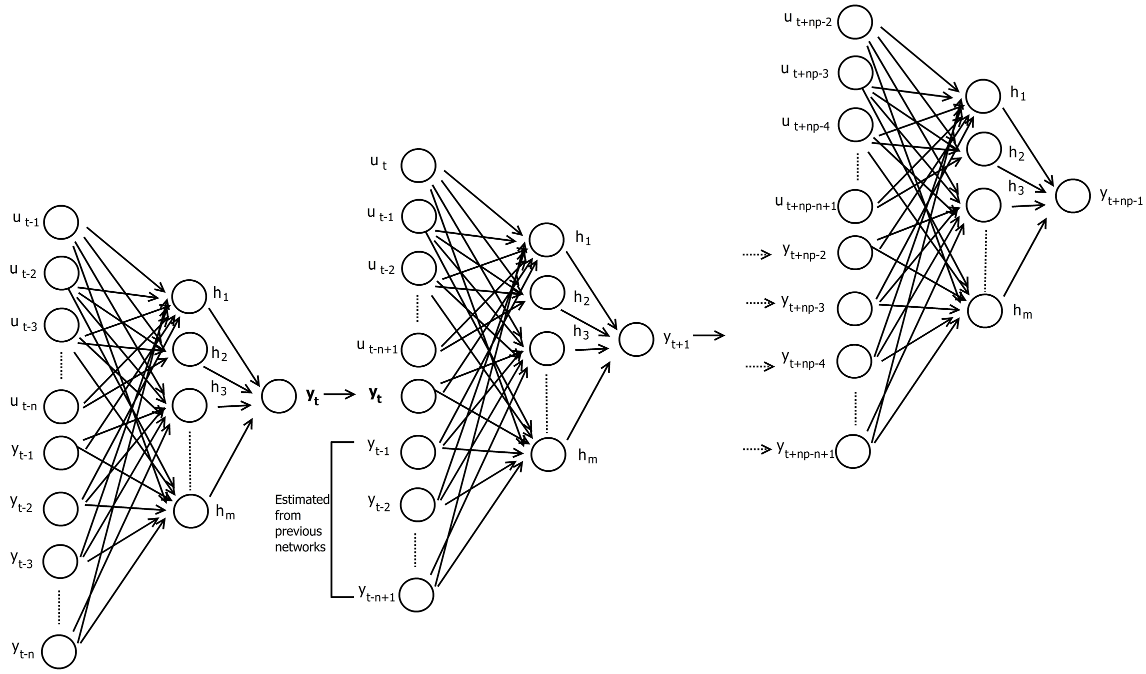


Figure 6.2: Dynamic neural network for MPC model

N_c number of inputs are obtained and among them the first value is used as the next time interval input and rest all are dumped. This prediction and optimization process works at every sampling intervals until the total running time of the process ends. The optimizer works by keeping the input and output values in a constrained range. These constraints can be the limits of hardware used in the process or fundamental limitations for example, mole fraction cannot be negative or higher than 1.

The model enclosed in the MPC can be a transfer function model, state-space model or nonlinear model such as neural network model, support vector regression model, etc. While the former two are time invariant linear models, they cannot be used for generalizing a process with high non linearity and dynamic in nature. In this case, a dynamic neural network model has been used in the MPC as shown in Fig. 6.2.

The dynamic neural network contains input layer including *n* number of previous input and output variables each, a hidden layer and output layer giving prediction of the future process output. This predicted output goes to next network and is used to estimate the output further until N_p^{th} output variable has been estimated. These N_p predictions goes to optimizer for the future N_c input variables estimation and among them 1^{st} estimated value goes to the plant and the rest are neglected. Since in the case of Recurrent Neural Network (RNN), the current output is dependent on previous output estimation, this network is also obeying same phenomenon and can be said to be dynamic neural network with recurrent characteristics. The optimizer enclosed in the MPC contains a *fmincon* optimization tool of MATLAB® for the optimization purpose. The *fmincon* is a constrained nonlinear programming solver which is a gradient based optimization technique that minimize the cost function by keeping the input and output variables withing the constrained limits.

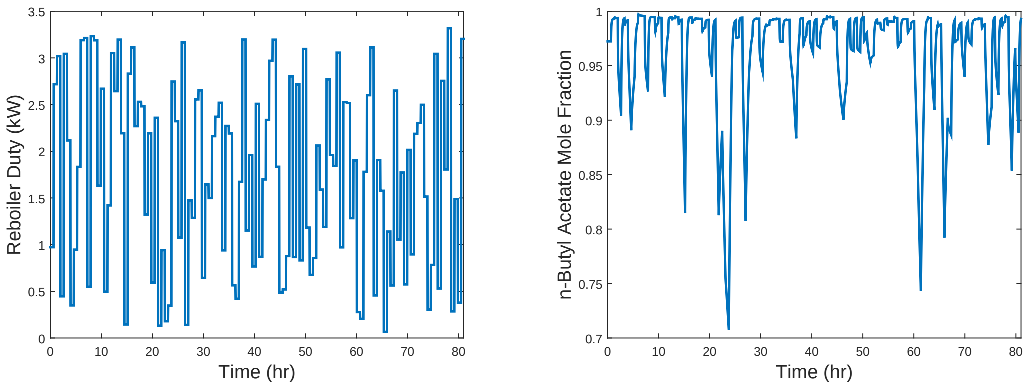
6.2 Results

The MPC contains two major components: a model and an optimizer. Both the component carries some parameters which needs to be optimized before using the MPC as a primary controller. These parameters involve number of neurons in neural network model, amount of historic data needed to be used for the prediction purpose, weights enclosed in the cost function, prediction horizon and cost horizon.

6.2.1 Dynamic Neural Network Model for MPC

An MPC works on the internal model of the process which needs to be accurate for the tighter control. Due to high complexity in RDC working, a dynamic neural network model was proposed for the purpose. The chemical plants are nonlinear and dynamic in nature, so the current model output cannot be estimated with only current input. The model has to follow previous input and output trend to give current process output. The amount of historical data needed for the current prediction depends upon the time taken by the process to get settled down at new steady state value when certain change in the input is provided to the process. It can be observed from Fig. 3.8 that the process is settling down to 90% of its final value at the time of 40 min after the step change was given to the model. As sampling interval taken in the process is 2 min, 20 historic sampling intervals are taken to relate the input and output of the process. Thus value of n in Fig. 6.2 which is the number of historic data that is required to estimate the current output of the process is 20.

The dynamic neural network model was developed using the sequential data which was collected by running the dynamic RDC model for around 81 hours. The data for the model was generated by giving random step changes in the reboiler duty which needs to remain constant for 20 sampling intervals and collecting the data of butyl acetate composition for the timed variations as shown in Fig. 6.3. The reboiler duty was varied by keeping it in the constrained range of 0.0267 to 3.33 kW, respectively. The data is used in the sequential form of the MATLAB® to keep its dynamic characteristics into the model. The 81 hours data was divided into the training data and testing data.



(a) (b)

Figure 6.3: Network training data (a) Reboiler Duty (b) Butyl Acetate bottoms composition

The training data contained first 60 h composition and reboiler duty data of the total data and the remaining last 21 h data is used for testing of the network. The network contained 40 input neurons containing a sequential data of previous 20 reboiler duties and 20 product composition data, 10 hidden neurons and 1 output neuron of future butyl acetate composition in the bottom product stream of RDC. When the network was trained and tested, it gave R^2 value of 1 during the training of network and 0.9998 during the testing of network. This perfect fit leads to the conclusion that the dynamic neural network model can be used as an internal model in the MPC.

6.2.2 MPC Parameters

MPC contains a model and an optimizer whose parameters need to be optimized before controlling the process. The parameters invloved in the control process are prediction horizon, *P*, control horizon, *C*, and cost function weights, W_y and $W_{\Delta U}$.

The *prediction horizon*, N_p, is the number of future samples of the process output which a model inside MPC has to predict and pass it to the optimizer for minimizing the cost function. *Cost horizon*, N_c, is the number of control moves the optimizer predicts by minimizing the cost function. It is assumed that after N_c moves the input of the process

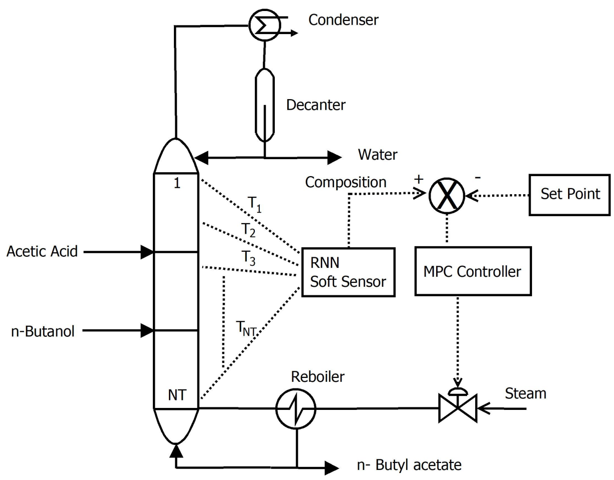


Figure 6.4: Reactive distillation column with RNN sensor and MPC

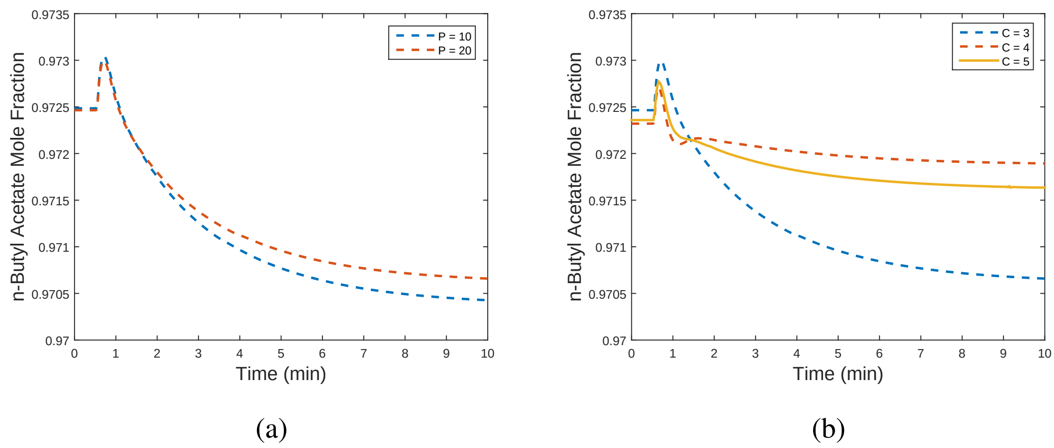


Figure 6.5: Effect of (a) Prediction horizon (b) Control horizon on closed loop with MPC

is constant. Both the horizon values are optimized by giving a step change in load variable in a closed loop with no integral action as shown in Fig.6.5. In both the figures, the set point is the initial value, the response closer to the set point is considered better for control purpose. It can be observed from Fig.6.5(a) that increasing the N_p value increases the response quality but the horizon was limited to 20 as after increasing it from the set value, it does not lead to any change in the response. The control horizon value is optimized to be 4 as the performance of controller degraded when the value is increased or decreased from this value. The cost function includes weights which are signifying the amount of role of output and input deviation in the control action. The value of both the weights W_y and $W_{\Delta U}$ were set to 1, increasing from this value leads

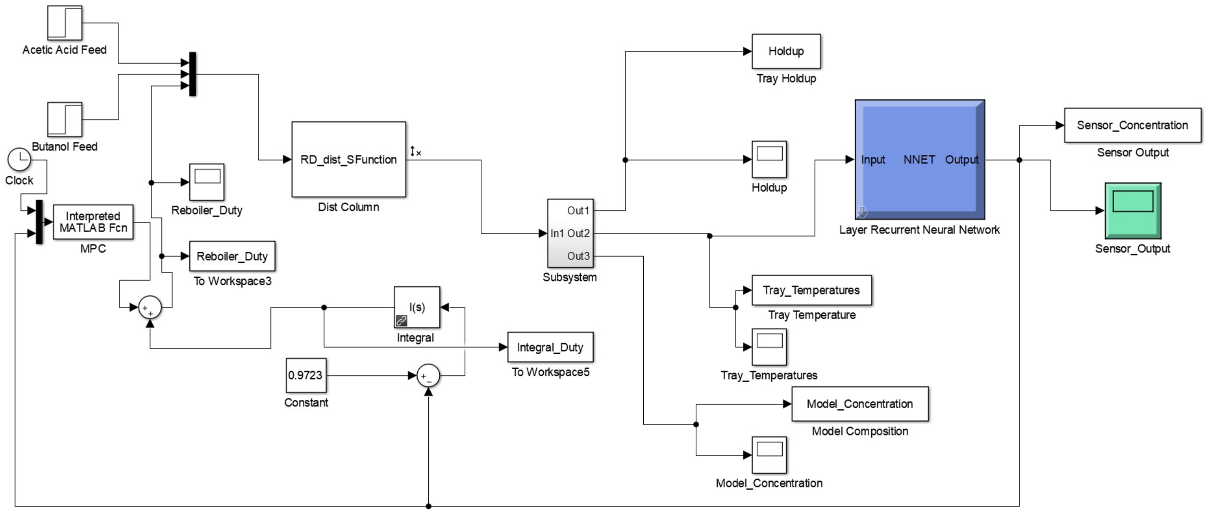


Figure 6.6: Closed loop SIMULINK® diagram with MPC controller

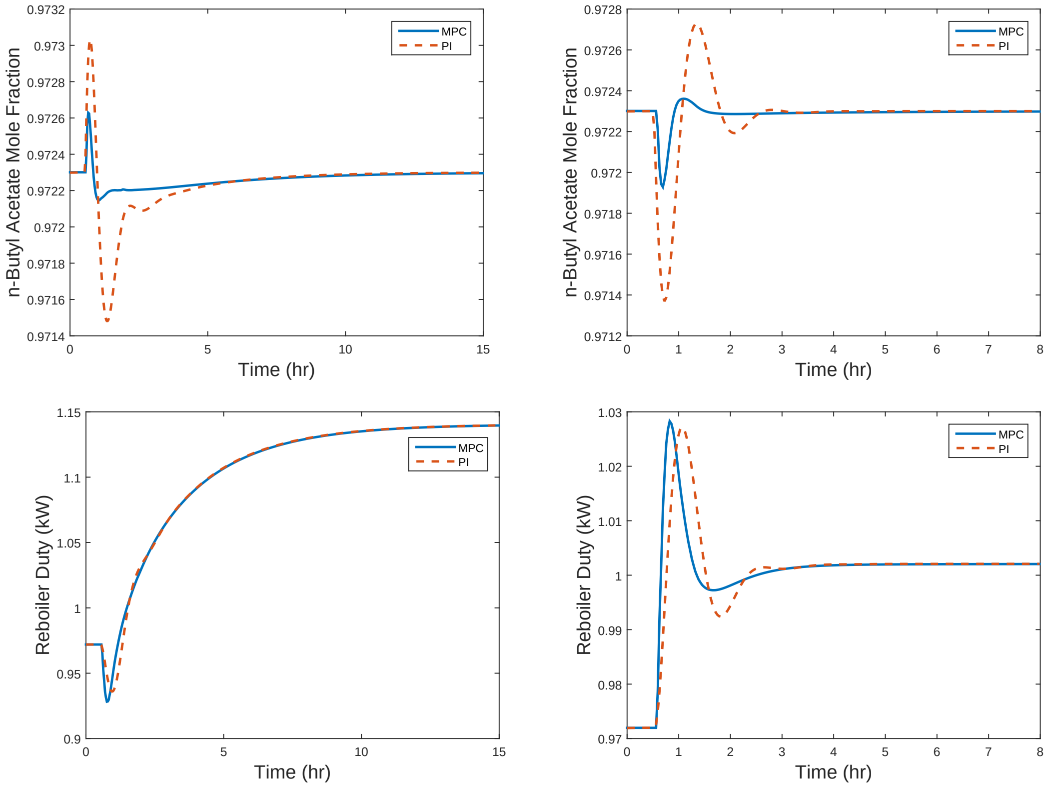
to no change in response and decrease leads to oscillatory response.

6.2.3 Closed Loop Response

Finally after developing a neural network model and optimizing the parameters of MPC, MPC coupled with an integral action was used to control the butyl acetate composition in the bottoms of RDC as shown in Fig.6.4. The integral action of τ_I = 90 hr is used to remove the offset from the response. The SIMULINK® diagram of the closed loop with MPC controller is shown in Fig. 6.6. The MPC block contains the code of MPC which is embedded in the SIMULINK® diagram using interpreted MATLAB® function. The MPC controlled closed loop has been compared with PI controlled closed loop having PI parameters of K_c = 4.61 and τ_I = 177.45 h tuned using MATLAB® auto tuning method. The product composition which is being controlled by MPC and PI was taken from RNN based soft sensor which is acting as a measuring element for the closed loop.

Table 6.1: Performance indexes for load change in Acetic Acid feed flow rate

Acetic Acid	ISE		IAE		ITAE	
Step Change	MPC	PI	MPC	PI	MPC	PI
-8%	5.69×10^{-8}	5.22×10^{-7}	6.46×10^{-4}	1.35×10^{-3}	2.41×10^{-3}	2.93×10^{-3}
+10%	2.18×10^{-8}	2.91×10^{-7}	1.40×10^{-4}	5.6×10^{-4}	2.07×10^{-4}	3.41×10^{-4}



(a) (b)

Figure 6.7: Closed loop response for load change in Acetic Acid flow rate by $(a) - 8\%$ $(b)10\%$

6.2.3.1 Load change in Acetic Acid feed flow rate

The RDC column was given a step change of -8% and $+10\%$ in the acetic acid feed rate and then controlled using both MPC and PI controllers. The step change was given after 30 min of process start. It can be observed from Fig. 6.7 that MPC is showing better results with less overshoot, less undershoot and less sluggish behavior than PI controller. The control results are also verified by controller performances as shown in Table 6.1. It can be observed that there is a significant decrease in all the integral errors while using MPC rather than PI.

6.2.3.2 Load change in Butanol feed flow rate

The regulatory response was also studied by giving step disturbances in butanol feed rate by -10% and $+8\%$. It can be seen in Fig. 6.8 that MPC response is substantially better than PI controller response. The MPC leads to decrease in the overshoot and oscillation

Table 6.2: Performance indexes for load change in Butanol feed flow rate

Butanol	ISE		IAE		ITAE	
Step Change	MPC	PI	MPC	PI	MPC	PI
-10%	1.33×10^{-7}	2.36×10^{-6}	3.11×10^{-4}	1.56×10^{-3}	5.60×10^{-4}	1.02×10^{-3}
+8%	3.90×10^{-7}	3.19×10^{-6}	1.36×10^{-3}	2.39×10^{-3}	3.34×10^{-3}	3.69×10^{-3}

to higher extent as compared to PI. The performance criteria as shown in Table 6.2, verifies the above conclusion of MPC being better than PI for regulatory response of the model.

Table 6.3: Performance indexes for set point change

Set Point	ISE		IAE		ITAE	
Step Change	MPC	PI	MPC	PI	MPC	PI
-0.01	6.57×10^{-6}	1.72×10^{-5}	1.18×10^{-3}	3.44×10^{-3}	6.44×10^{-4}	1.61×10^{-3}
+0.01	6.89×10^{-6}	1.73×10^{-5}	1.59×10^{-3}	3.17×10^{-3}	1.29×10^{-3}	1.31×10^{-3}

6.2.3.3 Set Point change

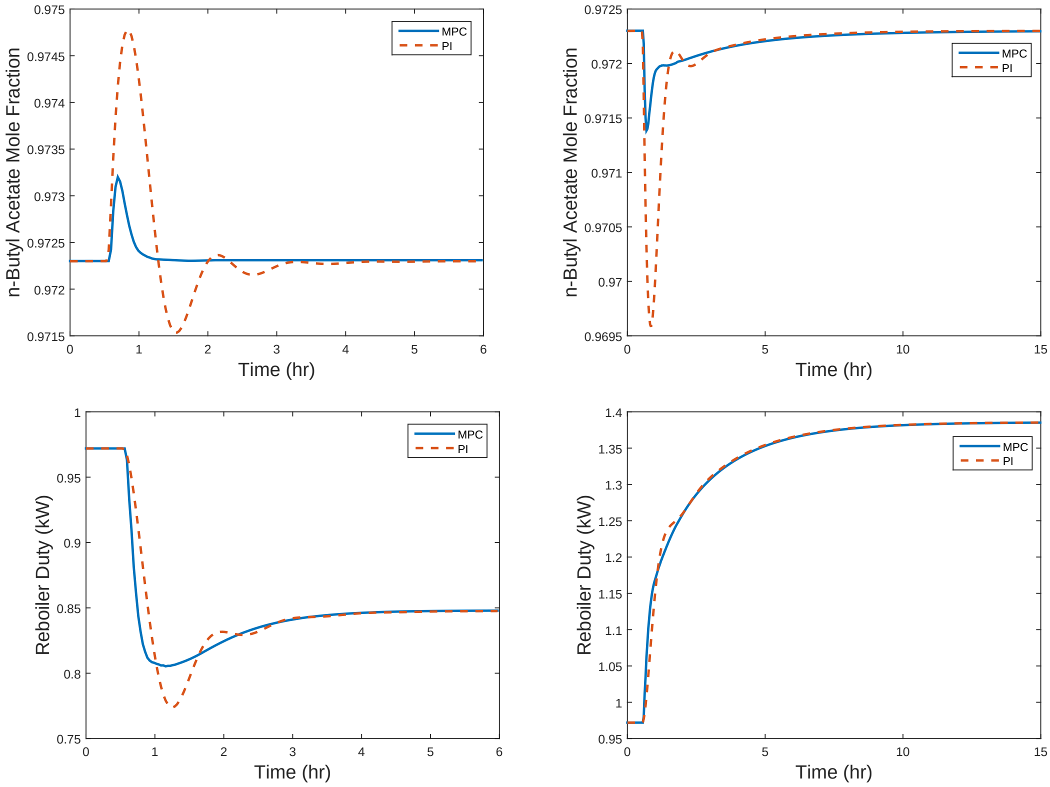
After regulatory control, RDC was also considered for servo control system. The RDC was given step change in the set point by ± 0.01. It can be observed from Fig. 6.9 that in the case of negative change, PI is controlling the process with more oscillations than MPC. An MPC is showing high oscillation in the manipulating variable during step change but it is getting settled quickly than PI. In the case of positive change, both the responses have a small difference but MPC provided comparatively better results with quick less sluggish behavior than PI. The integral errors are shown in Table. 6.3 and all the errors decrease for MPC.

6.3 Controller Performance in the Presence of PRBS Disturbance

As discussed earlier, PRBS is a more practical way to check a controller performance than step changes. A PRBS signal is provided for both the load variables to compare the performance of MPC controller with PI controller.

6.3.0.1 Load Change in Acetic Acid Feed Flow Rate

The regulatory response for the load change in acetic was studied by giving a disturbance in the form of a PRBS signal with the magnitude of $\pm 10\%$ of the steady state



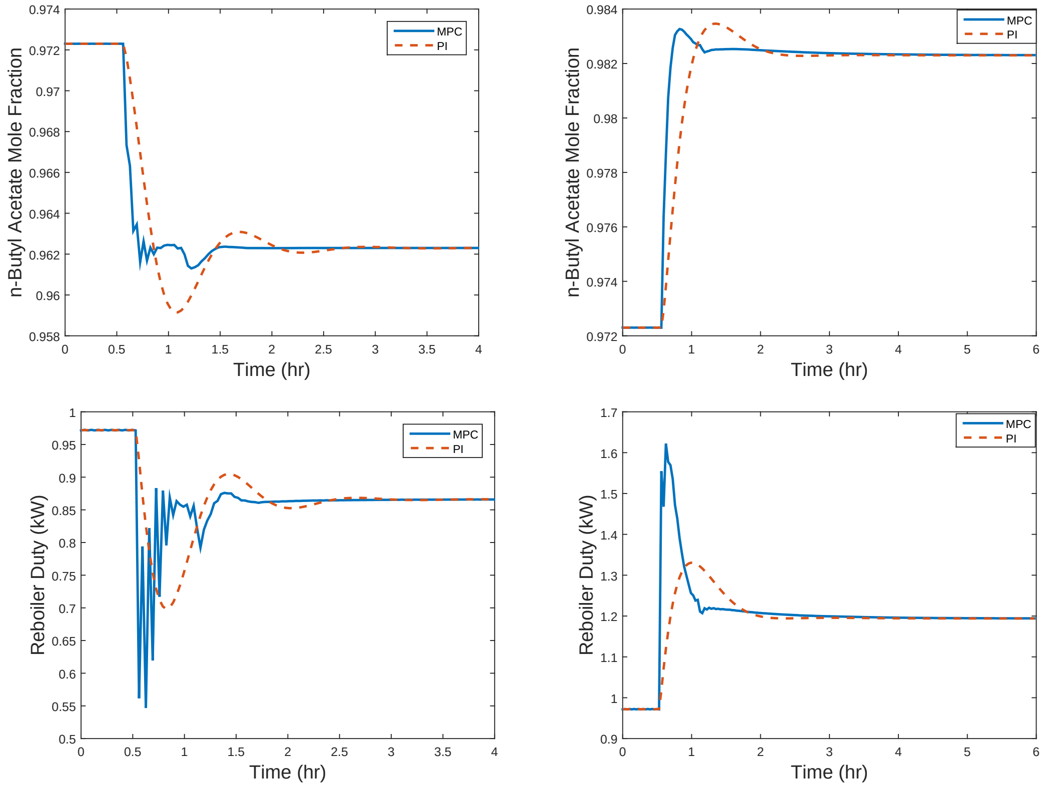
(a) (b)

Figure 6.8: Closed loop response for load change in Butanol flow rate by $(a) - 10\%$ $(b) + 8\%$

value of 10.5 mol/h. The PRBS signal is shown in in Fig. 6.10(c). It can be observed from Fig. 6.10(a) that at all the impulses, MPC is showing small overshoot than PI controller. The variation in the reboiler duty after addition of disturbance is shown in Fig. 6.10(b). The control results were also verified by controller performances as shown in Table 6.1. It can be observed that there is a significant decrease in all the integral errors while using MPC rather than PI. The computational time for calculation of optimized manipulated variable in each sampling interval of 2 min was approximately 50 seconds.

6.3.0.2 Load Change in Butanol Feed Flow Rate

The closed loop response was studied by giving a disturbance in butanol feed rate by $\pm 10\%$ in the form of PRBS signal. The steady state value of butanol feed rate is 9.5 mol/h. The closed loop composition and reboiler duty profiles are shown in Fig. 6.11 (a) & (b). The composition profile of butanol also followed the same trend of less overshoot



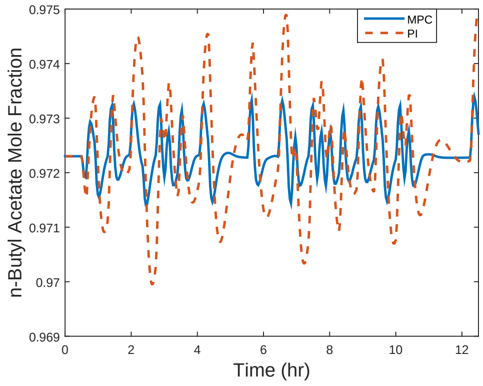
(a) (b)

Figure 6.9: Closed loop response for set point change by $(a) - 0.01\%$ $(b) + 0.01\%$

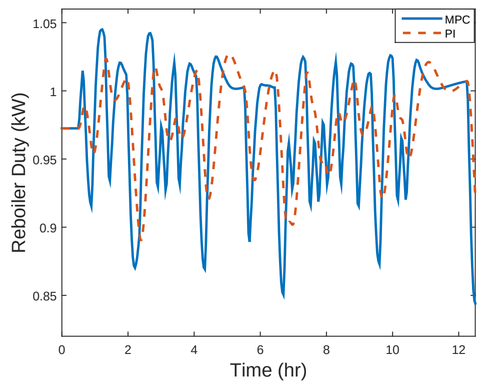
and high stability as it was shown above by acetic acid composition profile. The closed loop composition profile suggests that MPC performs better than PI controller for the PRBS disturbance as shown in Fig. 6.11 (c). The performance criteria as shown in Table 6.1 verifies the above conclusion of MPC being better than PI for PRBS signal disturbances too.

Table 6.4: Performance indexes for load change in Acetic Acid and Butanol feed flow rate

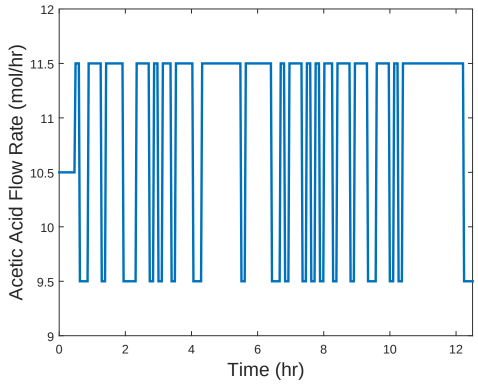
	ISE		IAE		ITAE	
Load	MPC	PI	MPC	PI	MPC	PI
Acetic Acid	2.30×10^{-6}	1.25×10^{-5}	3.93×10^{-3}	9.58×10^{-3}	2.18×10^{-2}	5.34×10^{-2}
Butanol	1.93×10^{-6}	3.75×10^{-6}	3.19×10^{-3}	5.22×10^{-3}	1.77×10^{-2}	2.89×10^{-2}



(a)



(b)



(c)

Figure 6.10: Closed loop response for load change in Acetic Acid flow rate (a) Composition profile (b) Reboiler duty profile (c) PRBS load change signal

Table 6.5: Performance indexes for load change in Acetic Acid feed flow rate for RDC with dead time

	$\tau_d = 1min$		$\tau_d = 3min$	
-8%	MPC	PI	MPC	PI
ISE	6.97×10^{-08}	5.65×10^{-07}	9.47×10^{-08}	6.90×10^{-07}
IAE	6.86×10^{-04}	1.35×10^{-03}	7.21×10^{-04}	1.39×10^{-03}
ITAE	2.39×10^{-03}	2.83×10^{-03}	2.38×10^{-03}	2.82×10^{-03}
$+10\%$	MPC	PI	MPC	PI
ISE	2.75×10^{-08}	3.33×10^{-07}	5.20×10^{-08}	4.53×10^{-07}
IAE	1.55×10^{-04}	6.08×10^{-04}	1.90×10^{-04}	7.47×10^{-04}
ITAE	2.35×10^{-04}	4.47×10^{-04}	2.63×10^{-04}	6.35×10^{-04}

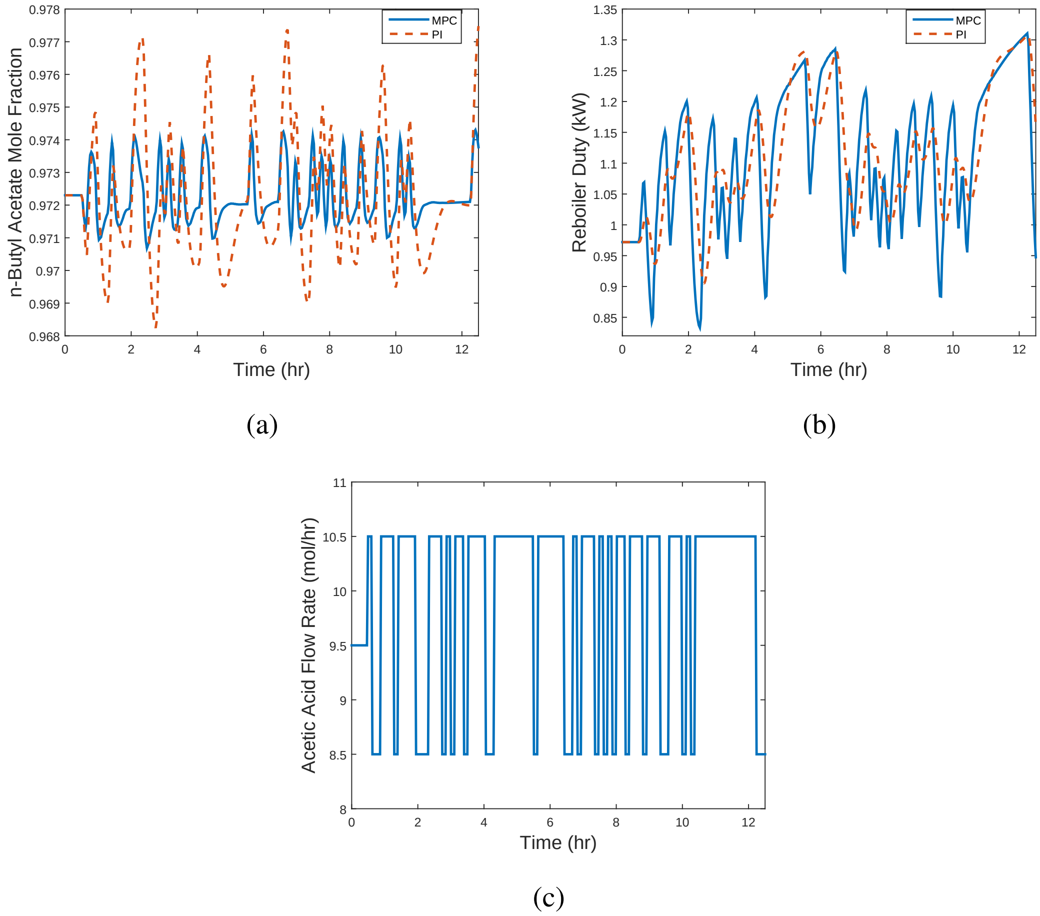


Figure 6.11: Closed loop response for load change in Butanol flow rate (a) Composition profile (b) Reboiler duty profile (c) PRBS load change signal

6.4 Process with Dead Time

The process plant always carries some time lag in the form of transport delay. The amount of delay varies upon the types of process, length of pipes, flow rate of fluids etc. In the case of reactive distillation, it has been mentioned in Luyben and YU (2008) that transport delay of 1 min is considered when tray temperature is controlled and 4-6 min is taken for the composition control. The high transport delay during composition control is because of the time lag taken by hardware sensors to estimate the composition. In the case of soft sensor, as the sensor is estimating the composition at a real time, 1 min transport delay can be considered for the study. But to study the effect of higher transport delay, 3 min transport delay is considered by assuming that this can be the maximum delay which a process can experience without having lag in measuring element. The SIMULINK® closed loop diagram of the RDC with transport delay is

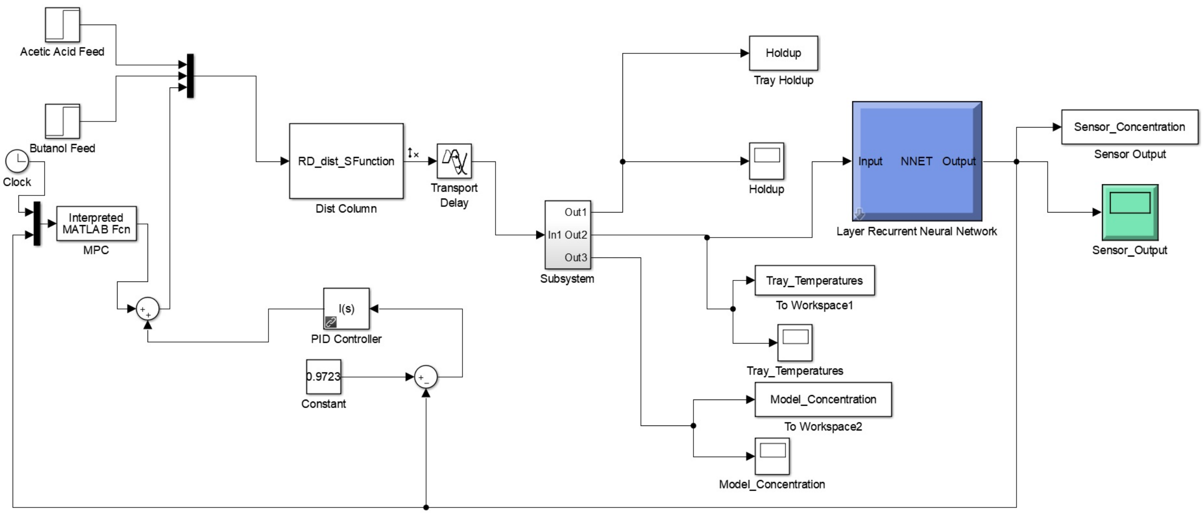


Figure 6.12: Closed loop SIMULINK® diagram of RDC with transport delay and MPC

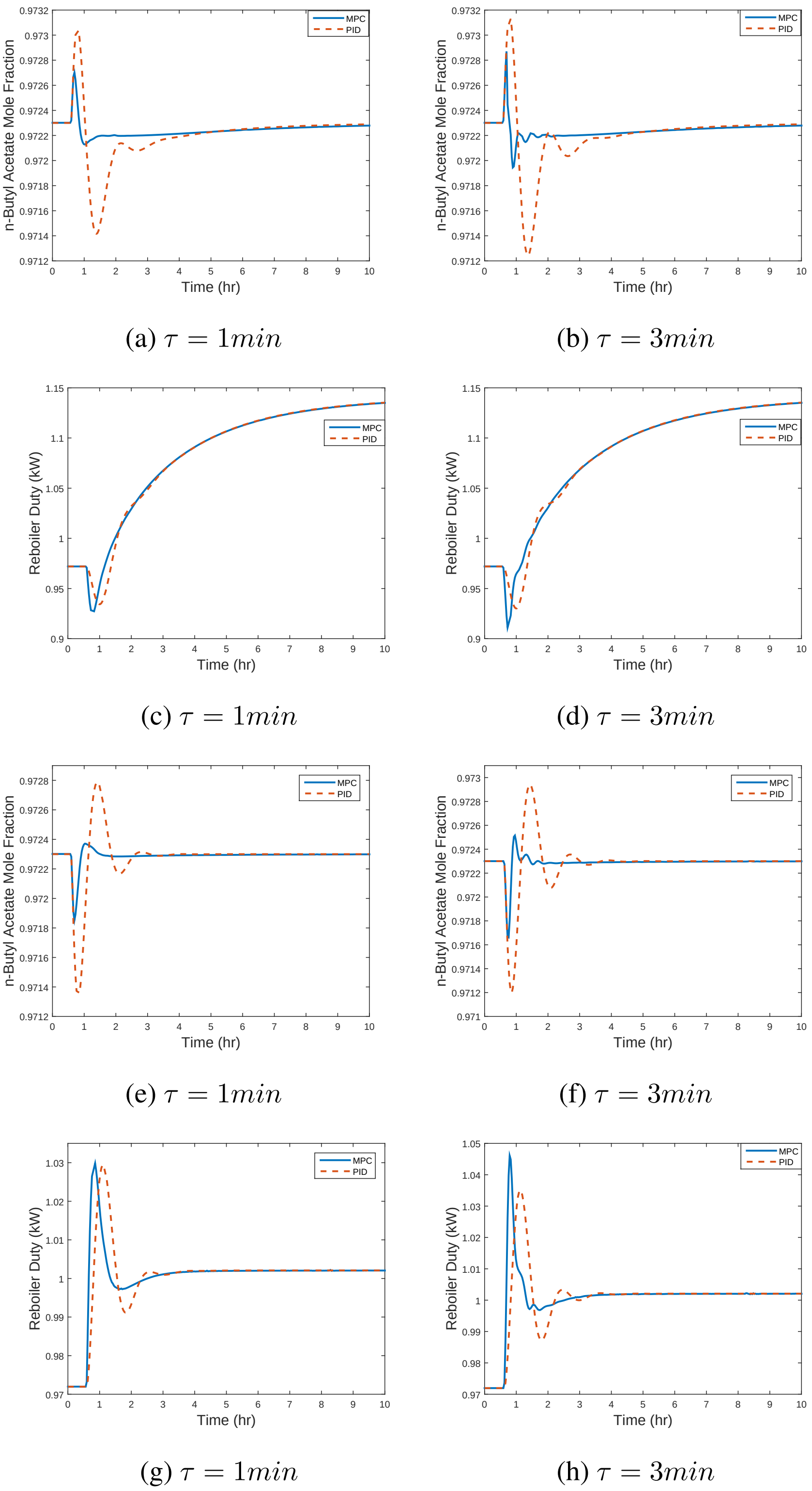
shown in Fig. 6.12. The block after S-function adds the transport delay in the process. To study the effect of dead time in the closed loop, servo and regulatory response has been studied for both the delays of 1 min and 3 min separately in the system.

6.4.1 Load Change in Acetic Acid Feed Flow Rate

The effect of dead time in the RDC model has been studied by giving the step change of -8% and $+10\%$ in the acetic acid feed rate. The composition and reboiler duty profile is shown in Fig. 6.13. It can be observed from the profiles that the model is showing oscillatory behavior when the process is having higher dead time of 3 min. In all the cases, the MPC for both the dead times is showing better results than PI controller with small overshoot, small undershoot and better stability towards oscillatory behavior of the model. All the integral errors are shown in Table. 6.5 . The integral errors are also less for every case of MPC when they are compared to errors of PI controller.

6.4.2 Load Change in Butanol Feed Flow Rate

The regulatory response was also studied by giving a step change of $+8\%$ and -10% in the butanol feed rate in the presence of dead times of 1 min and 3 min separately. The butyl acetate composition profile and RDC reboiler duty profile during the step changes is shown in Fig. 6.14. When the dead time is 1 min, the PI controller is showing small oscillations, while MPC is not showing any oscillatory behavior. When the dead time in the process is 3 min, both the controllers are showing oscillations, whereas



(a) $\tau = 1min$ (b) $\tau = 3min$

(c) $\tau = 1min$ (d) $\tau = 3min$

(e) $\tau = 1min$ (f) $\tau = 3min$

(g) $\tau = 1min$ (h) $\tau = 3min$

Figure 6.13: Closed loop response for load change in Acetic Acid flow rate composition profile for (a) & (b) -8% (e) & (f) $+10\%$; Reboiler duty profile for (c) & (d) -8% (g) & (h) $+10\%$

Table 6.6: Performance indexes for load change in Butanol feed flow rate for RDC with dead time

	τ_d= 1 min		τ_d= 3 min	
-10%	MPC	PI	MPC	PI
ISE	1.66×10^{-07}	2.61×10^{-06}	3.08×10^{-07}	3.48×10^{-06}
IAE	4.31×10^{-04}	1.67×10^{-03}	6.02×10^{-04}	2.29×10^{-03}
ITAE	1.16×10^{-03}	1.27×10^{-03}	1.29×10^{-03}	2.58×10^{-03}
$+8\%$	MPC	PI	MPC	PI
ISE	5.44×10^{-07}	3.79×10^{-06}	6.46×10^{-07}	4.39×10^{-06}
IAE	1.43×10^{-03}	2.41×10^{-03}	1.42×10^{-03}	2.43×10^{-03}
ITAE	2.82×10^{-03}	3.02×10^{-03}	2.81×10^{-03}	3.03×10^{-03}

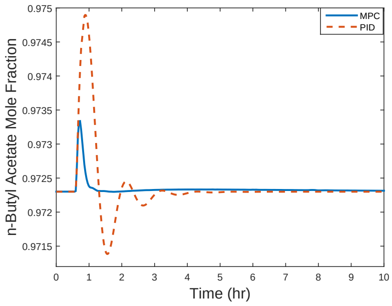
the oscillations are less in MPC as compared to PI. The other problems of overshoot, undershoot and sluggishness is also solved during the use of MPC in the case of this regulatory response. The integral errors as shown in Table 6.6 also shows the same scenario of small errors for MPC than PI.

Table 6.7: Performance indexes for set point change for RDC with dead time

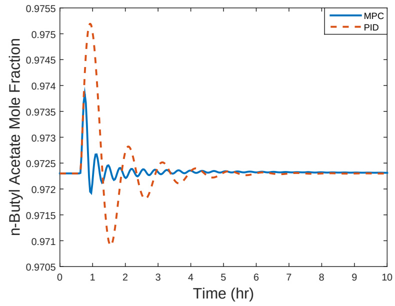
	τ_d = 1 min		τ_d= 3 min	
-0.01%	MPC	PI	MPC	PI
ISE	7.46×10^{-07}	5.65×10^{-07}	1.69×10^{-05}	2.76×10^{-05}
IAE	1.09×10^{-03}	3.87×10^{-03}	3.05×10^{-03}	5.46×10^{-03}
ITAE	2.09×10^{-04}	1.96×10^{-03}	1.83×10^{-03}	3.77×10^{-03}
$+0.01\%$	MPC	PI	MPC	PI
ISE	8.18×10^{-06}	1.86×10^{-05}	1.21×10^{-05}	2.17×10^{-05}
IAE	1.79×10^{-03}	3.32×10^{-03}	2.41×10^{-03}	3.76×10^{-03}
ITAE	1.19×10^{-03}	1.34×10^{-03}	1.86×10^{-03}	1.93×10^{-03}

6.4.3 Change in Set Point

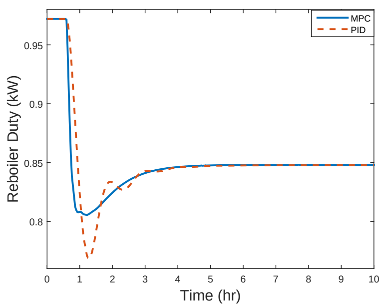
The closed loop stability during the presence of dead time was also studied for a servo control system problem. The servo response was analyzed by giving a step change of



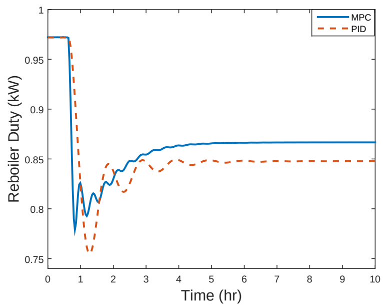
(a) $\tau = 1min$



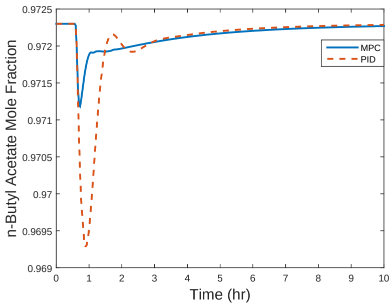
(b) $\tau = 3min$



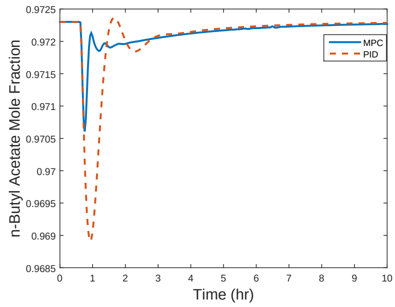
(c) $\tau = 1min$



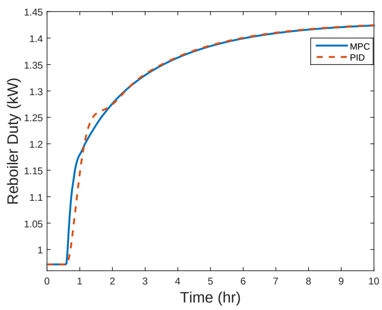
(d) $\tau = 3min$



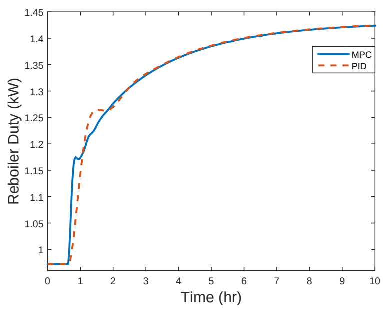
(e) $\tau = 1min$



(f) $\tau = 3min$

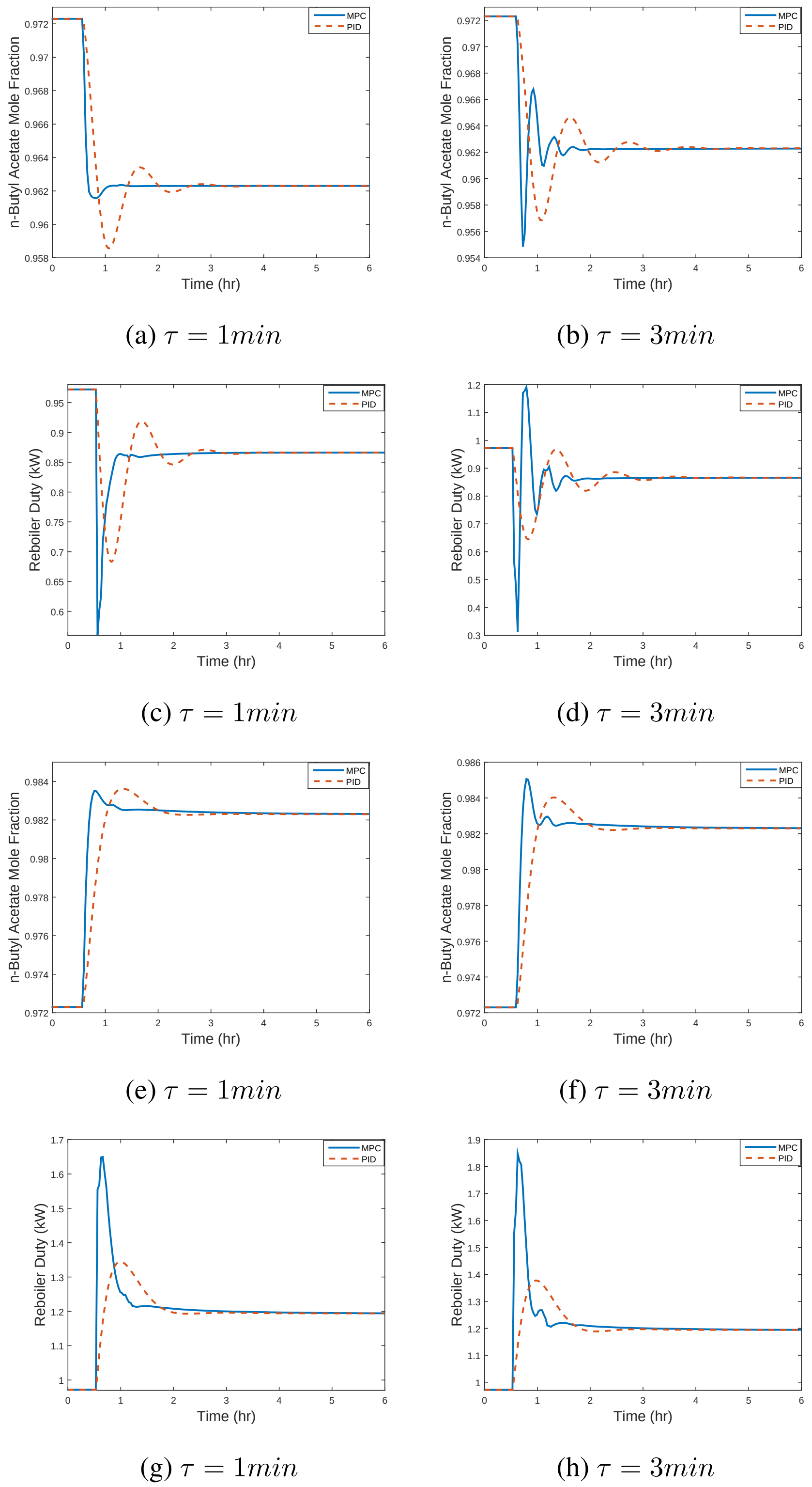


(g) $\tau = 1min$



(h) $\tau = 3min$

Figure 6.14: Closed loop response for load change in Butanol flow rate composition profile for (a) & (b) -10% (e) & (f) $+8\%$; Reboiler duty profile for (c) & (d) -10% (g) & (h) $+8\%$



(a) $\tau = 1min$ (b) $\tau = 3min$

(c) $\tau = 1min$ (d) $\tau = 3min$

(e) $\tau = 1min$ (f) $\tau = 3min$

(g) $\tau = 1min$ (h) $\tau = 3min$

Figure 6.15: Closed loop response for setpoint change rate composition profile for (a) & (b) -0.01% (e) & (f) $+0.01\%$; Reboiler duty profile for (c) & (d) -0.01% (g) & (h) $+0.01\%$

± 0.01 in the set point value of 0.9723. The composition and reboiler duty response during the set point change is shown in Fig. 6.15. It can be observed from the composition profile that dead time of 1 min is not affecting the MPC controller to high extent but there is some oscillations in the case of PI controlled closed loop. For the dead time of 3 min, both the controllers are showing high oscillations. The MPC is showing high overshoot during dead time of 3 min but the response is faster in the case of MPC rather than PI. The integral errors as shown in Table. 6.7 also suggest that the errors are not highly deviating in the case of τ_d = 3 min but MPC still shows small errors because of the faster response than PI. In the case of τ_d= 1 min all the errors decreases substantially while using MPC rather than PI.

6.5 Conclusions

In this chapter, the problem of inverse response due to load variable was handled using model predictive control (MPC) technique. The technique depends upon the future predictions of the model which leads to the optimization of process input while considering the problem of inverse response before it affects the process. The model inside the MPC was developed using dynamic neural network technique which can accommodate all the non linearities of the RDC. The closed loop response of the controller was studied by giving step and PRBS signal disturbances. In all the servo and regulatory control systems, the MPC proved to be the better controller than PI. The closed loop response of the MPC has lower overshoot, undershoot and fast response as compared to PI controlled RDC. The study was also extended by including the dead times of 1 min and 3 min into the process. In the presence of dead time the response became oscillatory but MPC gave better results in that case too. The controllers performance was weighted on the basis of integral errors. During both the cases of process with or without dead time, the MPC provided less integral errors than PI controller proving its superiority over PI.

Chapter 7

Conclusions and Recommendations

7.1 Conclusions

The estimation and control of *n*-butyl acetate composition in the bottom stream of reactive distillation column was considered for the present study. The open loop response of the RDC model observed the problem of inverse response for the step change in load variable as acetic acid feed flow rate. This inverse response causes high overshoots and sluggish response of the RDC. The problem of inverse response due to the load variable was removed using a feedforward-feedback controller for an RDC. The inverse response due to manipulated variable for a 3^{rd} order transfer function model was also solved using a compensator for the case study of an RDC. The problem of inverse response due to load variable was further solved using an MPC controller with an internal model based on dynamic neural network. The point-wise conclusion is summarized below:

1. A dynamic nonlinear mathematical model of an RDC for synthesis of *n*-butyl acetate using esterification reaction was implemented on MATLAB® software and solved using an ODE15s solver. The model involved the material balance, energy balance, and thermodynamic equilibrium equations at all the trays of the column along with the liquid-liquid equilibrium calculations for the decanter to separate the aqueous and organic phases from top of the column. The MATLAB® mathematical model was transformed into a dynamic simulation model in SIMULINK® software for further sensing and control studies.

2. The MATLAB® /SIMULINK® RDC model was validated with the experimental study of Steinigeweg (2002) and CHEMCAD® software results.

3. During the open loop study of the RDC model, it was observed that the column

was showing an inverse response for a step change in a load variable i.e. acetic acid feed rate. This inverse response due to the load variable was solved using a feedforward-feedback controller to control the product composition of the RDC using transfer functions of process and load disturbances. The feedforward-feedback controller results were compared with the closed loop having only feedback controller. The performance of both the controllers was evaluated using performance indexes such as ISE, IAE and ITAE. Since the feedforward-feedback controller is a model based technique, the robustness of the controller was tested by giving uncertainties in the parameters of the load and process transfer function model.

4. The feedforward-feedback controller gave better results for transfer function model of the RDC but when applied to plant model, the performance deteriorated for 8% increase in step change in acetic acid feed rate. The reason for the performance deterioration is the inability of transfer function model to cover all the non-linearities of the RDC.

5. The literature does not include the solution of inverse response due to manipulated variable for a 3^{rd} order transfer function model of the process. The problem of inverse response for a 3^{rd} order transfer function model of the RDC column was solved by designing a compensator for an inverse response and dead time compensation. The compensator performance was compared with the closed loop without a compensator and evaluated using performance indexes. The compensator robustness was also tested by giving model mismatch for the various values of gain and time constants of process transfer function model.

6. The soft sensor was developed using a recurrent neural network technique for the estimation of butyl acetate composition in the bottoms stream of RDC using tray temperatures of the column. The soft sensor performance was evaluated by comparing the sensor estimation and true value of composition for the open loop and closed loop study of the RDC. The soft sensor performance was estimated using mean square error (MSE) between sensor output and true composition value. The butyl acetate composition in bottom stream of RDC was controlled using a PI controller and RNN based soft sensor as a measuring element in the closed loop.

7. To solve the problem of inverse response due to step change in load variable in the RDC, another method of designing an MPC for the RDC was studied. The dynamic neural network technique was used to develop an internal model of MPC. The MPC parameters such as prediction horizon, control horizon, and weights of cost function were optimized by studying the closed loop of the RDC with MPC controller; the optimum values obtained are prediction horizon as 20, control horizon as 4, and weights of cost function, W_y and W_{DeltaU}, as 1 each . The problem was extended by including a transport delay of 1 min and 3 min in the process. The MPC controller was compared with a PI controller tuned using MATLAB auto tuning method with the parameters as K_c = 4.61 and τ_I = 177.45 h. The performance of both the controllers was evaluated using performance indexes. The computational time for calculation of optimized manipulated variable in each sampling interval of 2 min was approximately 50 seconds.

7.2 Recommendations

Some of the future work which can be done to extend the study is as follows.

1. The data which was collected from the MATLAB® /SIMULINK® model can be taken from the real plant to train more realistic soft sensor.

2. The soft sensor can be validated by estimating the product composition of an experimental study using the sensor.

3. The soft sensor code can be incorporated in a hardware and can be used as a hardware sensor.

4. Design the combination of dynamic sensor and controller for other process units such as reactors, or other complex distillation columns.

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BIO-DATA

Name	Gaurav Kataria
Father's Name	Ashok Kumar Kataria
Mother's Name	Anita Kataria
Date of Birth	08/08/1990
Marital Status	Unmarried
Educational Qualification	M.Tech. Chemical Engineering (2014) MNIT Jaipur B.Tech. Chemical Engineering (2012) NIT Jalandhar
Publications	International Journal (01-Published and 01-In communication) International Conference (06) National Conference (01)