SIMULATION OF MALEIC ANHYDRIDE PRODUCTION IN FIXED BED REACTOR

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2017

SIMULATION OF MALEIC ANHYDRIDE PRODUCTION IN FIXED BED REACTOR

by

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Thesis submitted in partial fulfilment of the requirement for the degree of Bachelor of Chemical Engineering

May 2017

ACKNOWLEDGEMENTS

This thesis would not have been possible without the inspiration and support of a number of wonderful individuals. My thanks and appreciation to all of them for being part of this journey and making this thesis possible. I owe my deepest gratitude to my supervisors Associate Professor Dr. Norashid Aziz. Without his enthusiasm, encouragement, support and continuous optimism this thesis would hardly have been completed. His guidance into the world of building information simulation has been a valuable input for this thesis. I also want to express my gratitude to my colleague Belinda Anne A/P Xavier and Fatin Nur Liyana Binti Mohd Idris. They has made available his support in a number of ways, especially towards the completion of this thesis.

Finally, my deep and sincere gratitude to my family for their continuous and unparalleled love, help and support. I am grateful to family for always being there for me as a friend. I am forever indebted to my parents for giving me the opportunities and experiences that have made me who I am. They selflessly encouraged me to explore new directions in life and seek my own destiny. This journey would not have been possible if not for them, and I dedicate this milestone to them.

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LIST OF SYMBOL

Symbol	Description	Unit
D	Diameter or reactor	m
d_p	Diameter of particle	m
ε	Void fraction	-
E_i	Activation energy	kJ/mol
F_t	Feed total	m^3/h
k_1	Rate constant	m^3/kg
L	Length of reactor	m
R	Gas constant	J/mol K
T_{inlet}	Temperature inlet	°C

LIST OF ABBREVIATIONS

MAN	Maleic anhydride
US	United state
VPO	Vanadium phosphorus oxide

SIMULASI PRODUKSI MALEIK ANHIDRIDA DIDALAM REAKTOR LAPISAN TETAP

ABSTRAK

Di dalam kajian ini, simulasi pengoksidaan separa n-butana telah dibangunkan. Aspen Plus V8.8 telah digunakan untuk merangsang, menganalisa, mengoptimumkan proses pengoksidaan separa n-butana dan oksigen. N-butana dimasukkan ke dalam RPLUG model bersamaan 0.0075 pecahan mol dan 0.21 pecahan mol oksigen. Suhu dan tekanan bagi penyuapan aliran di programkan kepada 379.85°C dan 1.6 atm. Diameter untuk reactor adalah 0.0025m dan panjang 1.0m. Pemankin yang digunakan dalam simulasi ini adalah vanadium phosphorus teroksida. Simulasi di dalam Aspen model telah dibandingkan dengan sorotan kajian untuk mengesahkannya. Ujian sensitif telah dijalankan dengan tiga kondisi yang berbeza iaitu suhu, tekanan dan kadar aliran suapan. Dalam pengoptimum, fungsi objektif adalah untuk memaksimumkan pertukaran n-butana. Pengoptimum memberi nilai optimum iaitu kadar suapan n-butana adalah 0.001 kmol/jam dan suhu reaktor pada 550°C.

SIMULATION OF MALEIC ANHYDRIDE PRODUCTION IN FIXED BED REACTOR

ABSTRACT

In this study, simulation of partial oxidation n-butane was developed. Aspen Plus V8.8 was used for stimulated, analysed, and optimized process partial oxidation of n-butane and oxygen. N-butane feed to RPLUG model is equal to 0.0075 mole fraction and 0.21 mole fraction of oxygen which is rich oxygen state. Temperature and pressure of feed streams are set at 379.85°C and 1.6 atm. Diameter for reactor is 0.0025 m and length with 1.0 m. Catalyst used in the simulation is vanadium phosphorus oxide. Simulation of Aspen model was compare with literature to validate. Sensitivity was carried out with three different condition which is temperature, pressure, and feed flow rate. In optimization, the objective function is to maximize conversion of n-butane. The optimization give optimum value which is molar flow rate n-butane in at 0.001 kmol/hr and reactor temperature at 550°C.

CHAPTER ONE

INTRODUCTION

1.1 Research background

Maleic anhydride (MAN) is one of an intermediate product to produce unsaturated polyester resin and demands for industry annual worldwide with consumption of 2.7 Mt. (Trifirò and Grasselli, 2014). Other products that are being manufactured from maleic anhydride including paper sizing resins, insecticides and fine chemicals. It is a solid substance appearing as colourless needles or white crystalline solid. Maleic anhydride is moderately soluble in water and hydrolyses upon contact with water to maleic acid. Approximately 70% of manufacturer using n-butane as their feedstock and remaining use benzene (Schunk, 2008). Vanadium phosphorus oxide (VPO) used as catalyst to convert maleic anhydride from partial oxidation of n-butane(Shekari and Patience, 2010).

Apparently, the catalyst performance declines with time as its selectivity decreases with simultaneously increasing activity. As a consequence the maleic anhydride yield diminishes and the reactor operation shifts towards less stable regions. Because of the general assumption that a loss of phosphorus from the catalyst is responsible for this unwanted behaviour, a variety of technologies are claimed compensating this loss in order to ensure optimum operation of commercial reactors (Burnett et al., 1987).

Selective oxidation of n-butane to maleic anhydride (MAN) over vanadium phosphorus oxide catalysts (VPO) is the only industrial process involving the selective oxidation of a light alkane. It is commonly recognized that the selective oxidation of n-butane proceeds according to a redox mechanism with lattice oxygen participation (Xiao et al., 2001).

Side reaction for partial oxidation n-butane will produce carbon dioxide and carbon monoxide. Carbon dioxide and carbon monoxide are formed by parallel and consecutive reactions, according to a triangular reaction network (Wellauer et al., 1986).

1.2 Problem statement

Partial oxidation of n-butane to maleic anhydride have attracted many researcher to improve process toward more economical. Demand of maleic anhydride grown up fast and yield of maleic anhydride is low. Simulation plays a key role in process optimizations as it offers a cost-effective. Simulation needed to enhance yield maleic anhydride. Temperature, pressure and n-butane feed flow rate play important role toward maleic anhydride production. Effect of temperature, pressure and n-butane is related toward catalyst. Catalyst is one of important part in the reaction. Yield of maleic anhydride also depend on catalyst. So, simulation is conducted to investigate effect of temperature, pressure and n-butane toward catalyst to produce maleic anhydride.

1.3 Research objectives

This research aims,

- To stimulate fixed bed reactor regarding of the production of the maleic anhydride.
- To validate between aspen data and literature data.
- To sensitivity analysis the parameter that will affect the maleic anhydride production which is pressure, temperature and n-butane feed flow rate.
- To optimize simulation in fixed bed reactor to achieve high yield of maleic anhydride.

CHAPTER TWO

LITERATURE REVIEW

2.1 Application of maleic anhydride

Maleic anhydride one of the monomer that can make unsaturated polyester resin. Polyester have their own important and most versatile polymers and suitable to use in many industry such as automotive industry, plastic industry, biomedical industry and many more. Anhydride or dicarboxylic acids will react with dihydroxy compound through polycondesation process to produce polyester (Rogers and Long). Maleic anhydride one of the most important monomer for introducing unsaturated in the polyester chain. Unsaturated polyester resins was developed in 1930's, and many industries still use it until now. In two or three years back, unsaturated polyester resin more favourable to biomedical application. Figure 2.1 show polycondensation reaction:

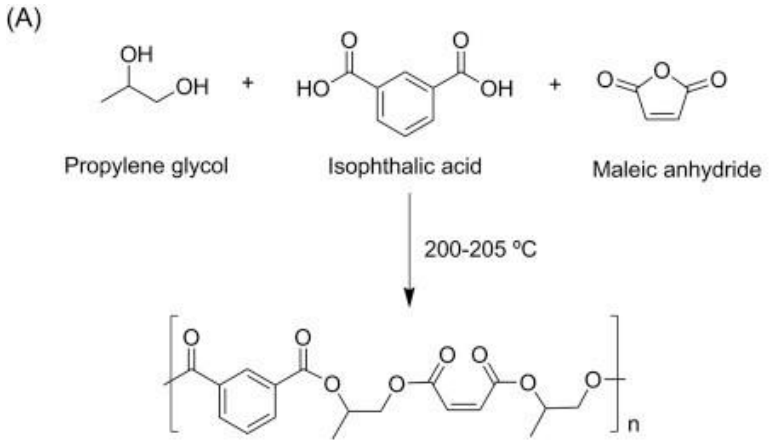

Figure 2.1: Polycondensation

2.2 Current industrial process

Huntsman was the largest manufacturer and accounted for over 9% of the total revenue in 2015. Huntsman corporation plant shown in Figure 2.2:

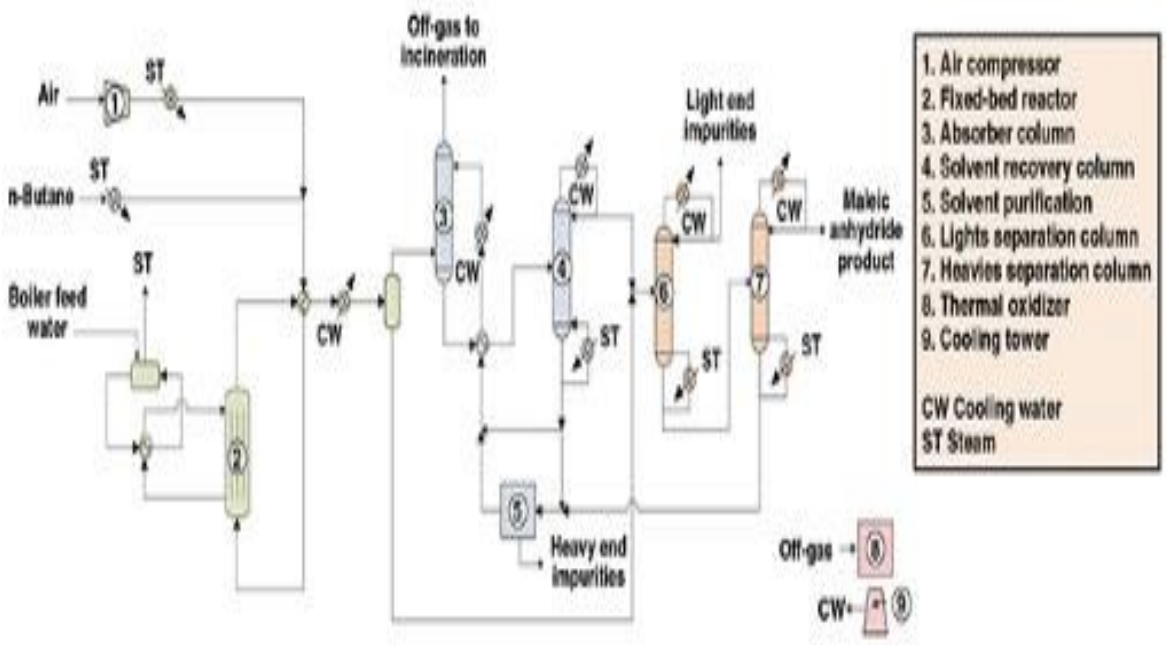

Figure 2.2: Huntsman plant overview

N-butane is vaporized and mixed with air before enter to reactor. Fixed bed reactor consists of multi tubular reactor with containing vanadium phosphorus oxide catalyst.

Heat generated is highly exothermic and it is removed by molten salt with jacketed around the reactor. Reactor effluent consist maleic anhydride, water vapour, carbon dioxide, carbon monoxide, and unconverted butane (Solutions, 2015).

2.3 Types of Reactor

2.3.1 Fixed bed reactor

The reactor that can be used to produced maleic anhydride which is fixed bed reactor. Fixed bed reactor normally runs at 80-85% n-butane conversion and overall yield of maleic anhydride is 57-65%. Partial oxidation of n-butane usually carried out in multi tubular fixed bed reactors filled with shaped catalyst particles. Reactor can include around 30,000 tubes of 21–25 mm diameter and 3–6 m length (Lesser et al., 2016).

Fixed bed is common technology where main concern to improve performances current depend on modification of catalyst rather than in reactor design (Dente et al., 2003).

2.3.2 Fluidised bed reactor

Fluidised- bed reactors usually used for large scale industry (Arnold et al., 1985). Fluidised bed technology represent newer and more complex technology. Most of the industry like BP and Mitsubishi, whereas ABB Lummus/Lorenza known as ALMA process. Its can operate at temperature below 450 °C and pressure of 2-4 bar. The feed composition of n-butane about 4-5 % v/v. The conversion of fluidised bed reactor exceeds 80%, but Maleic Anhydride yield just limited to 50-55%.

New Technology such as fluidised bed reactor to produce maleic anhydride in two bed system. It consist of riser reactor which vanadium phosphorus oxide catalyst, along some supplementary air, and oxidizes n-butane to maleic anhydride. Fluidised bed regenerator in which catalyst is reoxidized (Contractor, 1999). Fluidised bed reactor has advantage that can make higher butane conversion but it also has disadvantage which process requires attrition- resistance catalyst and circulates large amount of catalyst.

2.4 Partial oxidation of n-butane

Fixed bed reactor will n-butane and oxygen as feedstock and reaction occur at 450K (Ali and Al-Humaizi, 2014). N-butane and oxygen will mixed into fixed bed reactor with vanadium phosphorus oxide (VPO) catalyst. After the reaction occur, maleic anhydride will produce as main product. Carbon dioxide, carbon monoxide and water as side product.

$$C_4H_{10} + 3.5O_2 \xrightarrow{k_1} C_4H_2O_3 + 4H_2O \quad (2.1)$$

$$C_4H_{10} + 5.5O_2 \xrightarrow{k_2} 2CO + 2CO_2 + 5H_2O \quad (2.2)$$

$$C_4H_2O_3 + O_2 \xrightarrow{k_3} 4CO + H_2O \quad (2.3)$$

The excessive reaction heat are removed using molten salt. Excessive heat are removed in order to avoid run away temperature.

2.5 Reaction mechanism

The oxidation of the butane hydrocarbons to maleic anhydride is carried out by transfer of catalyst lattice oxygen species to the hydrocarbon. The lattice is then replenished by oxygen uptake from the gas phase.

For cyclic route (butadiene, dihydrofuran and furan) are found in a side route, they are assumed to be unstable on the catalyst surface. Therefore, they desorb more readily and are detected in the gas phase during n-butane oxidation. Besides desorption the cyclic species also can be reverted back to the corresponding alkoxide species by ring cleavage. The total oxidation of maleic anhydride involves the formation of carboxylic surface species. Figure 2.3 show reaction mechanism of n-butane:

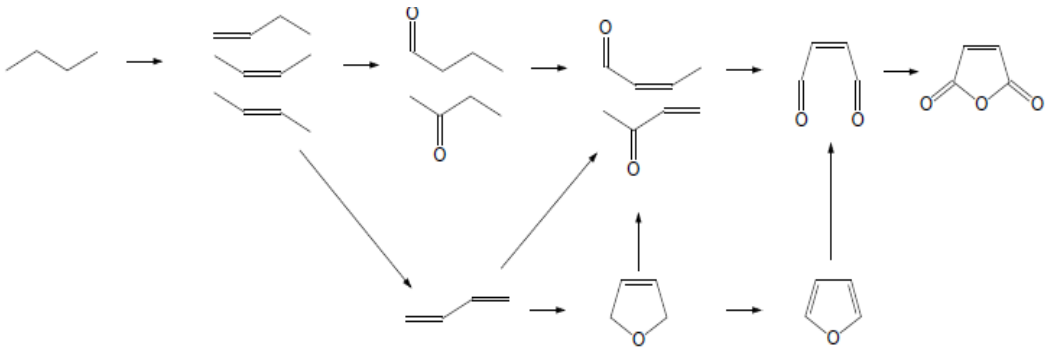

Figure 2.3: Reaction mechanism of n-butane

The Figure 2.4 shows the mechanism of partial oxidation of butane by using VPO catalyst. (Cornils, 2004) The number in figure indicates the reaction barrier and activation energy.

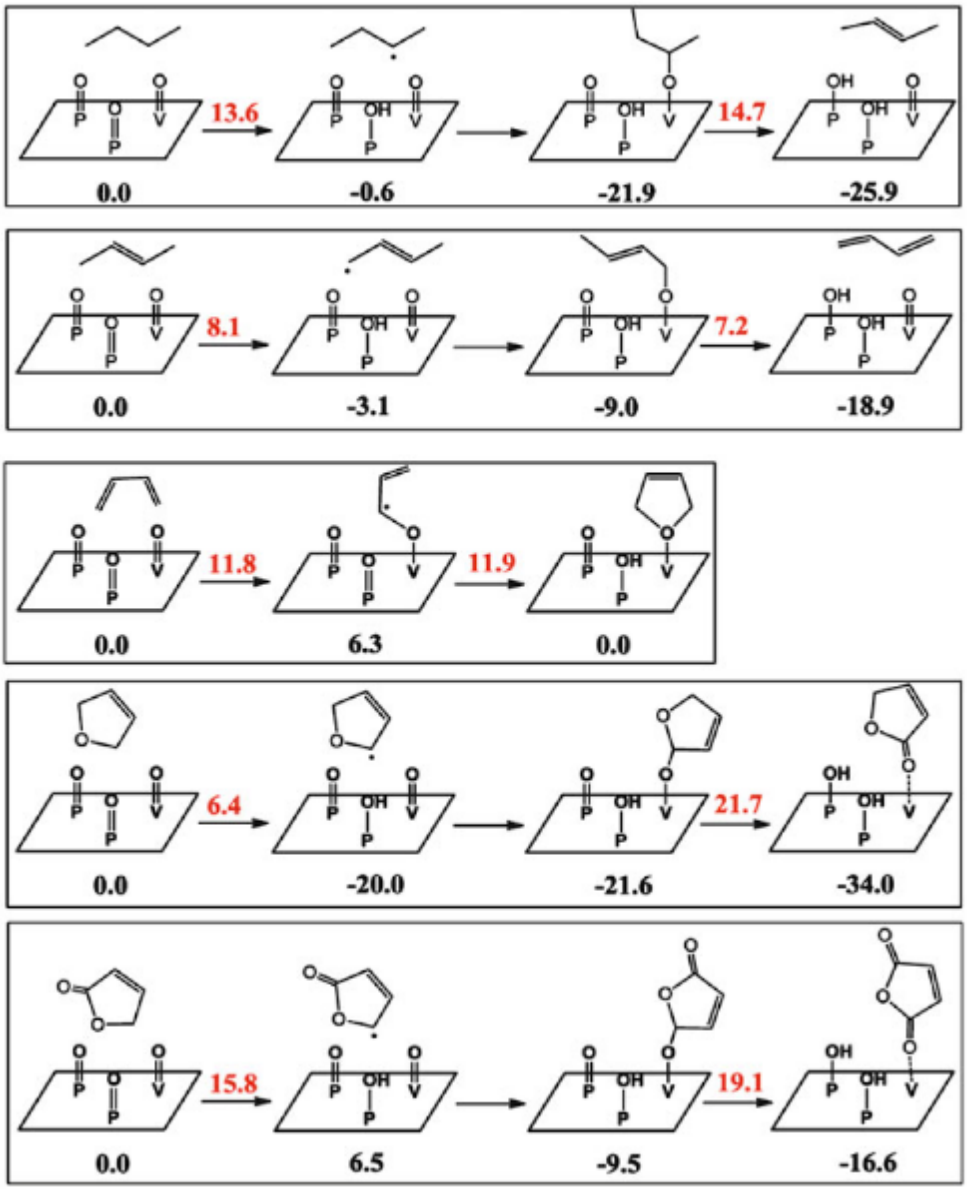

Figure 2.4: Reaction mechanism toward catalyst

2.5.1 Triangular Reaction Network

A simplified triangular reaction network is adopted together with the desired heterogeneous reaction of n-butane to maleic anhydride (Reaction 1), an undesired homogeneous oxidation reaction is assumed to occur in parallel (Reaction 2). Finally, the catalytic partial combustion of maleic anhydride closes the network (Reaction 3). (de Lasa et al., 2015). The triangular was illustrate as Figure 2.5:

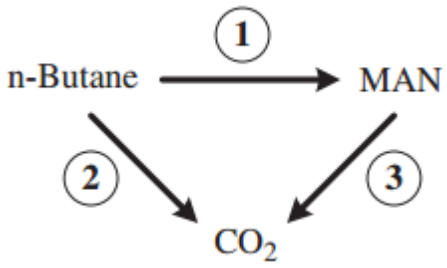

Figure 2.5: Triangular network of process

2.5.2 Reaction kinetic

Partial oxidation of n-butane consist of complex heterogeneously catalysed reaction. A summary of reaction kinetic can be found in a journal.

Redox model proposed by Mars van Krevelen can be best reaction mechanism (M. Alonso et al., 2001). This model accounts reaction inhibition by maleic anhydride and butane. Model equation as shown below:

$$r_1 = \frac{k_1 Cbut}{1 + K_1\left(\frac{Cbut}{C0_2}\right) + K_2\left(\frac{Cman}{CO_2}\right)} \quad (2.4)$$

$$r_2 = \frac{k_2\, Cbut}{1 + K_1\left(\frac{Cbut}{C0_2}\right) + K_2\left(\frac{Cman}{CO_2}\right)} \quad (2.5)$$

$$r_3 = \frac{k_3 Cbut}{1 + K_1\left(\frac{Cbut}{C0_2}\right) + K_2\left(\frac{Cman}{CO_2}\right)} \quad (2.6)$$

ki is rate constant and it calculated normalise reference temperature of 653 K

$$k_i = k_{i,653} \exp\left[\frac{-E_{a,j}}{R}\left(\frac{1}{T} - \frac{1}{653}\right)\right] \quad (2.7)$$

Reaction kinetic that use in this study is power law method.

2.6 Simulation Study

Xiao et al. (2001) study about temperature programmed oxidation in thermogravimetric (TGA) unit. The experiment objective to examine effect of temperature on reoxidation of the catalyst. Fixed bed micro reactor used to investigate time average performance. Then, experiment of both composition and temperature modulation were carried out in tubular fixed bed reactor. The result for modulation for both composition and temperature need at least temperature of 500°C for oxidation process to proceed rapidly and it might improve reactor performance.

Huang et al. (2002) study behavior of catalyst which used vanadium phosphorus oxide(VPO) catalyst. The experiment for identifying structure of reaction network, temporal separation experiments of reduction and oxidation of catalyst, transient response experiments and forced unsteady state operation of fixed bed reactor. So, for result of experiment at steady state reaction at 372°C , 402°C and 440°C and feed with 15% oxygen, 3% n-butane and remaining is helium. Then the major product ware found that carbon oxides, water, and maleic anhydride and trace side product like butane, acetic acid, acylic acid and furan was found. At temperature 402°C with poor oxygen supply which is 5% oxygen and 12% n-butane, side product traced is 2,5-dihydrofuran and methylbenzene. Experiment was conclude that surface

lattice oxygen and absorbed oxygen are directly response for formation of maleic anhydride and CO_2.

Ali et al. (2015) explained about optimal oxygen feeding policies to maximize production of maleic anhydride. In this study, two reactor configuration were examined which is cross flow reactor with 4 discrete feeding point and conventional packed bed reactor. In order to operate CFR in dynamic mode and to optimize multiple feed dosage, nonlinear predictive Controller (NLMPC) was used as optimal controller to enhance Maleic Anhydride yield. For packed bed reactor configuration, single oxygen feed was used. The result shows maximization maleic anhydride mole fraction to 0.002 and yield of 26%.

Dong et al. (2016) study about simulation of effect catalyst pore structure on fixed bed reactor performance. Camsol Multiphysics was use to do all simulation in this study. The 3-D reactor was simplified to 2-D rectangular geometry that assuming rotational symmetry. The result with high porosity of random packing in vicinity of the wall and no-slip boundary condition applied at the wall. The result of reactor performance in regard to changing pore diameters reveal that bi-modal catalyst with larger transport pore and higher surface area are preferred for given kinetics.

Ali and Al-Humaizi (2014) study about comparative study between experiment and simulation by using cross flow reactor. The objective of simulation to optimize oxygen usage by different strategies of oxygen dosage.For simulation part, power law used as kinetic reaction. Result that experimental data fit very well with relative error not exceed 5%. Large increase reaction temperature may lead to formation of hot spots which could damage catalyst or reactor. The selectivity of desire intermediate product, maleic anhydride, could be increased in cross flow reactor when different location stages oxygen feeding.

Diedenhoven et al. (2012) study about phosphorus dynamic toward catalyst vanadium phosphorus oxide. The phosphorus content was determine by reversible sorption of process. If no phosphorus is added to the reactant feed, the catalytic activity increases until runaway occurs. With addition of a proper amount of phosphorus, the loss can be compensated while excessive phosphorus addition results in complete catalyst deactivation. Adjusting the model parameters to experimental data may result in a model that can be used to optimize the performance of maleic anhydride reactors.

Xiao-Feng et al. (2002) study about transient kinetics of n-butane oxidation to maleic anhydride over a vanadium phosphorus catalyst. Reactor system operated under steady state mode to improve performance of catalyst packed and transient reaction kinetic is needed. The result of experiment was the role of lattice and adsorbed oxygen in the reaction process. A transient kinetic model taking account oxygen storage and diffusion in bulk phase.

2.6.1 Simulation study summary

The summary of simulation study show in Table 2.1 :

Table 2.1: Simulation study summary

Raw material	Simulation/experiment	Conditions	Comments	References
n-butane	Experiment	Composition and temperature modulation	Suitable temperature at least 500°C	Xiao et al. (2001)
	Experiment	Temperature 372°C , 402°C, 440°C, feed with 15% oxygen, 3% n-butane	Study about catalyst(reduction and oxidation)	Huang et al. (2002)
	Simulation	4 stages of feeding oxygen	Maximize yield MAN to 26%	Ali et al. (2015)
	Simulation	No slip boundary	Effect of catalyst pore toward reaction kinetic	Dong et al. (2016)
	Simulation	Different strategies of oxygen dosage	To optimize usage of oxygen	Ali and Al-Humaizi (2014)
	Simulation	Different phosphorus content in catalyst	To maximize yield of maleic anhydride	Diedenhoven et al., 2012)
	Experiment	Different feed of butane concentration	To study lattice oxygen of catalyst toward maleic anhydride	Xiao-Feng et al. (2002)

2.7 Aspen Model

In aspen plus RPlug is used to simulate partial oxidation of n-butane. Reaction kinetic must be known in order to simulate this process. Data collected from Ali and Al-Humaizi (2014) was plug in into Rplug aspen model. N-butane will feed to reactor and it will convert to maleic anhydride, water, carbon dioxide, and carbon monoxide.

CHAPTER THREE

METHODOLOGY

For objective in chapter one to achieve, process model was developed in Aspen Plus version 8.8. To developed process model a general flow shown in Figure 3.1:

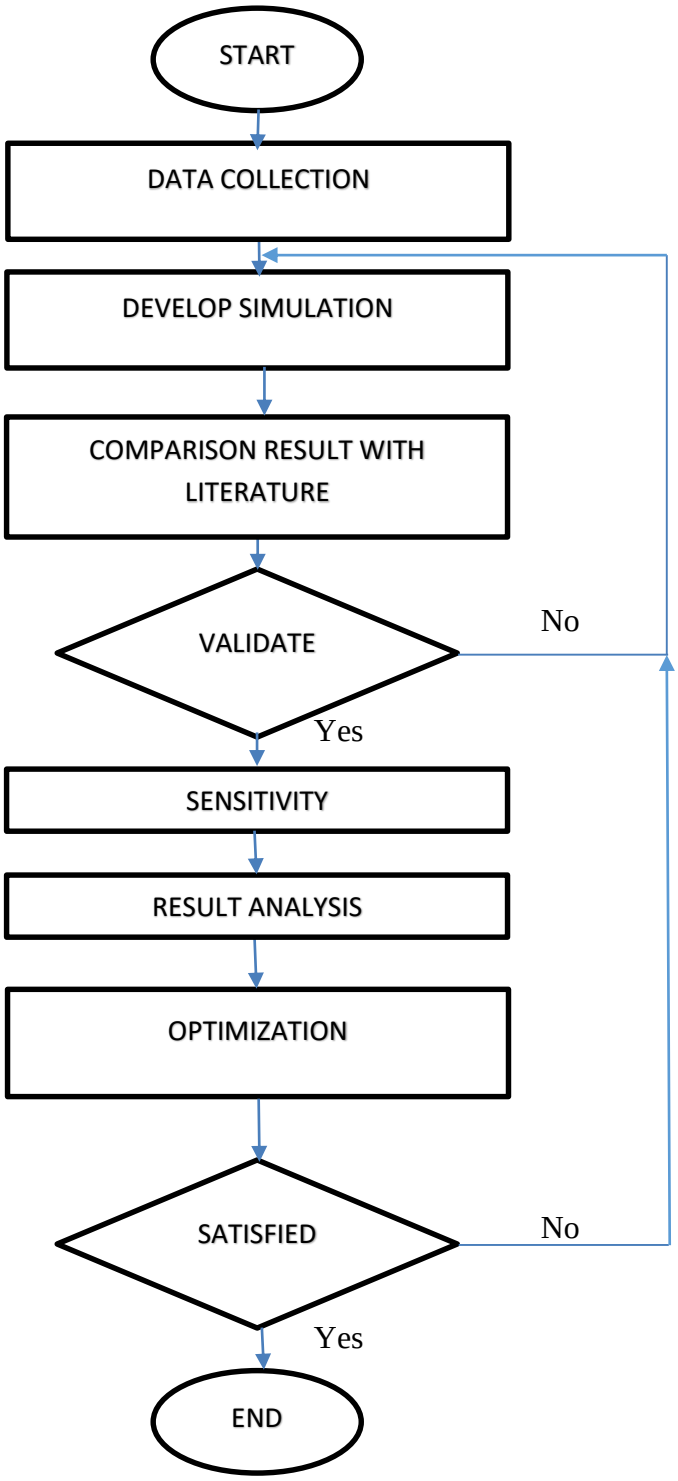

Figure 3.1: Flow diagram of methodology

3.1 Data collection

Aspen PLUS version 8.8 was used to predict the performance of the process. It was used to investigate the effect or different parameter toward maleic anhydride production. PFR was chosen because it involve reaction kinetic and catalyst in reaction. Data are collected from Ali and Al-Humaizi (2014) and summarized as Table 3.1 as below :

Table 3.1: Collected data from journal

Parameter	Value	Units	References
D	25×10^{-3}	m	(Sharma et al., 1991)
d_p	3×10^{-3}	m	
L	1.0	m	
ε	0.44	-	
$k_{1,653K}$	0.86	m^3/kg s	(M. Alonso et al., 2001)
$k_{2,653K}$	0.11	m^3/kg s	
$k_{3,653K}$	0.19	m^3/kg s	
E_1=E_2	80	kJ/mol	
E_3	98	kJ/mol	

Operating conditions

Parameter	Value	Units	References
T_{inlet}	177	°C	(Sharma et al., 1991)
F_t	1.65	m^3/h	
P	1.6	atm	
$Y_{C4,inlet}$	0.0075	-	
$Y_{O2,inlet}$	0.21	-	
$Y_{CO,inlet.}$ $Y_{CO2,inlet.}$ $Y_{MA,inlet}$	0	-	

Kinetic reaction use in this simulation is power law method as shown below:

$$k_i = k_{i,653} \exp\left[\frac{-E_{a,j}}{R}\left(\frac{1}{T} - \frac{1}{653}\right)\right] \quad (3.1)$$

3.2 Develop simulation

Literature information from Ali and Al-Humaizi (2014) was extract to run simulation. The input was put into aspen plus V8.8. Key in the information like composition, temperature, pressure in the aspen plus. Reaction kinetic must be specified in order to make reaction in reactor. Reactor parameter was key in into Rplug model. The method that has been decided to use in this simulation is Peng-Rob (Peng Robinson thermodynamic properties). The reactor using in aspen is RPlug which is reflect fixed bed rector.

3.3 Validate

The data will check after aspen has run. It will check to verify whether data from aspen deviate or not from literature review .In order to check whether simulation can proceed to further study by check error. The simulation can be proceed when error less or equal to 5%.

3.4 Sensitivity

Data from simulation will go through sensitivity. Sensitivity analysis was conducted to investigate the effect of parameter toward n-butane conversion and yield maleic anhydride. Reactor temperature, reactor pressure, and feed flow rate were considered as input variables for sensitivity analysis. The range for operating variables for sensitivity analysis shown in Table 3.2:

Table 3.2: Range for operating variables for sensitivity analysis

Parameter	Lower range	Upper range	Increment
Pressure (atm)	1	2.5	0.1
Temperature(°C)	300	550	50
N-butane feed flow rate (kmol/hr)	0.0004	0.004	0.000375

3.5 Optimization

Simulation will proceed to optimization. In simulation, parameter will be different in order to optimize some circumstances. Some parameter might be constant and some might be varies. The variable will be checked whether it follow the literature or not. Then, by changing the parameter, the result will be checked. Tool in aspen plus V8.8 will be used to optimize the process.

3.6 Improve

In this section, the data from the previous simulation will be exported. Then, the data will be comparing each other to see whether the optimization was successful or not. The results from the previous steps has been calculated. If the results shows that the data have a great improvement, the result will be accepted.

CHAPTER FOUR

RESULT AND DISCUSSION

4.1 Introduction

Chapter four mainly focus in two section. Firstly, comparison model prediction from result obtained in plug flow reactor in Aspen Plus and compared to simulation data from literature (Ali and Al-Humaizi, 2014).Secondly, Aspen Plus model is used to perform sensitivity analysis and to investigate effect of operating condition toward yield maleic anhydride and conversion n-butane. Lastly, result from sensitivity analysis will be used to carry out optimization. Optimization will evaluate optimum yield of maleic anhydride.

4.2 Aspen simulation result

The prediction of model were tested using simulation data by comparing maleic anhydride produced and oxygen consumed across reactor length. The model prediction from simulation Aspen Plus and journal well fit when it not exceed relative error 5%.

4.2.1 Effect of maleic anhydride produced and oxygen consumed toward reactor length.

Based on study, effect of maleic anhydride produced will increased across the reactor. Comparison with Ali and Al-Humaizi (2014) and simulated data for maleic anhydride produced in Figure 4.1:

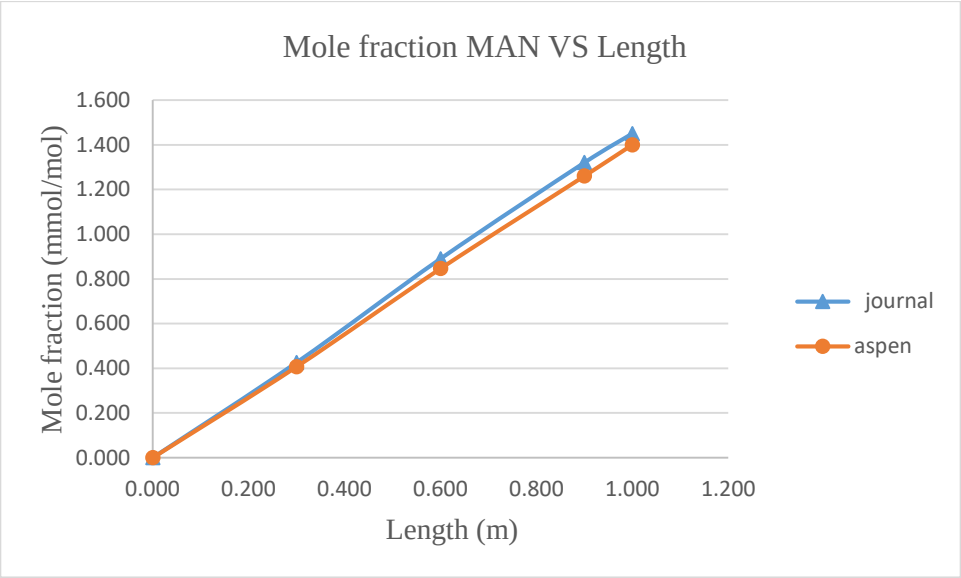

Figure 4.1: Comparison maleic anhydride simulated from literature and simulated Aspen

From the literature and simulated data for maleic anhydride produced, error was calculated for model prediction. Error was shown in Table 4.1:

Table 4.1: Calculated error for maleic anhydride produced in literature simulation Aspen.

Length (m)	Journal	Aspen	error
0.000	0.000	0.000	0
0.300	0.426	0.407	4.460
0.600	0.890	0.846	4.944
0.900	1.320	1.260	4.545
1.000	1.450	1.400	3.448

Effect of oxygen consumed will decreased across the reactor. Comparison literature and simulated data for oxygen consumed in Figure 4.2:

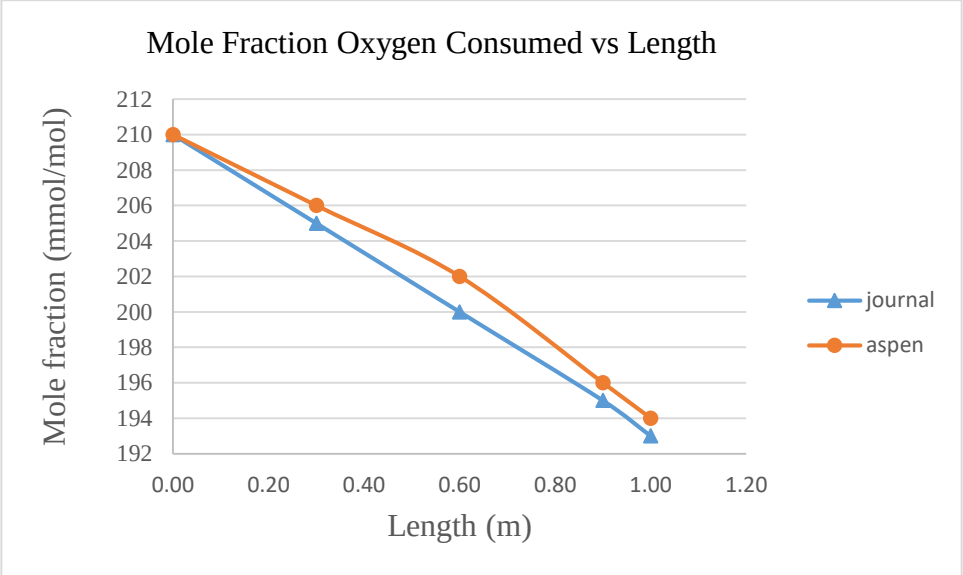

Figure 4.2: Comparison oxygen from literature and simulated Aspen

From the literature and simulated data for oxygen consumed, error was calculated for model prediction. Error was shown in Table 4.2:

Table 4.2: Calculated error for oxygen consumed in literature simulation Aspen.

Length (m)	Journal	Aspen	Error
0.00	210.00	210.00	0.00
0.30	205.00	206.00	-0.49
0.60	200.00	202.00	-1.00
0.90	195.00	196.00	-0.51
1.00	193.00	194.00	-0.52

4.2.2 Comparison of simulated results and literature data

From figure 4.1 both trend from Ali and Al-Humaizi (2014) and simulated Aspen Plus almost same. The trend from figure 4.1 increase linearly for mole fraction maleic anhydride from 0 to 1 meter of reactor length. The result obtained in Aspen Plus were correlated with data from literature. When length of reactor increase, mole fraction of maleic anhydride increased from 0 to 1.4 mmol/mol.

Furthermore, from figure 4.2 above, it clearly show amount of oxygen is decreased across the reactor length. The trend from literature and simulated Aspen Plus almost same. Mole fraction of oxygen, decreased from 210 mmol/mol to 194mmol/mol.

The trend for maleic anhydride increased and trend for oxygen decreased because exothermic reaction occur in the reactor. Feed reactant which is n-butane and oxygen will occur partial oxidation which is exothermic reaction. Maleic anhydride will keep increasing as it product of process and oxygen will keep decreasing as it reactant of the process. So that why, maleic anhydride will increased and oxygen will consumed across reactor length.

From Table 4.1 and Table 4.2 it shows that error occur for literature and simulated Aspen Plus. Amount of oxygen in Aspen Plus consumed less than literature. So it can be relate that amount of maleic anhydride produce from Aspen Plus is less than literature. The highest error about 4.944% that less than allowed error which is 5%.