SIMULATION AND OPTIMIZATION OF BIODIESEL PRODUCTION FROM DIMETHYL CARBONATE IN BATCH REACTOR

by

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LIST OF SYMBOLS

		Unit
CONV	Conversion	%
CONV*	Maximum Conversion	%
E_a	Activation Energy	kcal/mol
k_o	Pre-exponential Factor	sec^{-1}
M_{DMC}	Mole of DMC	mol
M_{PO}	Mole of Palm Oil	mol
Mss_{PO_0}	Initial Mass of Palm Oil	kg
$Mss_{PO\eta}$	Final Mass of Palm Oil	kg
η	Number of Time Intervals	-
P	Pressure	atm
R^2	Mean Square Error	-
t	Time Variable	sec
tb	Batch Time	sec
tb*	Minimum Batch Time	sec
T_{Fi}	Initial Feed Temperature	°C
T_R	Reactor Temperature	°C
T_{RL}	Lower Reactor Temperature	°C
T_{RU}	Upper Reactor Temperature	°C
V_S	Volume of Solution	L
V_{SL}	Lower Volume of Solution	L
V_{SU}	Upper Volume of Solution	L

LIST OF ABBREVIATIONS

CO_2	Carbon Dioxide
CSTR	Continuous Stirred Tank Reactor
DMC	Dimethyl Carbonate
ER	Experimental Result
FAGCs	Fatty Glycerol Carbonates
GC	Glycerol Carbonate
GDC	Glycerol Dicarbonate
H_2O	Water
H_2SO_4	Sulphuric Acid
KOH	Potassium Hydroxide
LCA	Life Cycle Assessment
MeOH	Methanol
NaOH	Sodium Hydroxide
NLP	Nonlinear Programming
OP	Optimization Problem
PFR	Plug Flow Reactor
PME	Palm Methyl Ester
RSM	Response Surface Methodology
SQP	Sequential Quadratic Programming
SR	Simulation Result
TG	Triglyceride / Triolein
TGA	Thermogravimetric Analysis
UNIQUAC	Universal Quasi-Chemical

SIMULASI DAN PENGOPTIMUNAN BAGI PENGHASILAN BIODIESEL DARIPADA DIMETIL KARBONAT DALAM REAKTOR KELOMPOK

ABSTRAK

Simulasi bagi penghasilan biodiesel daripada dimetil karbonat (DMC) adalah sangat sukar dan terhad terhadap penggunaan minyak kanola sebagai bahan mentah. Dalam kajian ini, proses DMC transesterifikasi pada skala perintis dalam penghasilan biodiesel dengan menggunakan reaktor kelompok telah diambil kira. Pengoperasian reaktor kelompok berskala perintis telah diperkenalkan dalam penghasilan biodiesel sebagai satu persediaan untuk pembangunan komersial dan industri. Simulasi reaktor kelompok berskala perintis bagi tindak balas DMC transesterifikasi menggunakan Aspen Plus versi 2006 telah diperkenalkan dalam kajian ini.

Model simulasi dipastikan kesahihannya dengan keputusan eksperiment yang terdapat dalam literasi. Model simulasi itu disahkan pada suhu 75°C dengan R^2 = 0.96 dan menghasilkan trend profil kepekatan PME yang sama dengan keputusan eksperimen. Berdasarkan model simulasi yang sah, reaktor kelompok skala perintis telah dilakukan dan kajian kepekaan telah dijalankan untuk mengkaji perilaku proses DMC transesterifikasi pada skala yang lebih besar. Reaktor kelompok berskala perintis dengan isipadu 300 L diperkenalkan dengan faktor 660 dari skala eksperiment. Berdasarkan kepada kajian kepekaan, suhu reaktor dan bilangan minyak kelapa sawit dipilih sebagai pembolehubah yang akan dimanipulasikan dalam kajian pengoptimunan.

Kajian pengoptimunan telah dijalankan dengan tiga objektif iaitu maksimum penukaran (OP1), minimum masa kelompok (OP2) dan maksimum kepala sawit metil ester (PME) dan minimum sisa buangan secara serentak (OP3). Kajian

pengoptimunan tersebut telah dilaksanakan menggunakan alat pengoptimunan dalam Aspen Plus iaitu berdasarkan kepada teknik pengaturcaraan kuadratik berurutan (SQP). Keputusan menunjukkan bahawa bagi OP1, penukaran tindak balas DMC transesterifikasi telah meningkat daripada 90.6% kepada 99.9%. Bagi OP2, masa kelompok yang diperlukan untuk tindak balas tersebut telah dipendekkan daripada 8 jam kepada 2.68 jam manakala bagi OP3, penghasilan PME telah meningkat dengan ketara daripada 31.05 kg kepada 125.99 kg dan sisa buangan telah berkurang daripada 76.42 kg kepada 63.78 kg pada masa kelompok yang singkat.

Dalam realiti, pengoperasian suhu ke atas reaktor kelompok mengalami perubahan dengan masa. Oleh itu, daripada mengoptimumkan suhu yang tetap, pengoptimunan profil suhu juga diambil kira. Dengan mengoptimumkan profil suhu, keputusan prestasi yang diperolehi bagi proses DMC transesterifikasi adalah lebih baik. Penghasilan PME bertambah manakala masa kelompok telah dipendekkan dan penghasilan bilangan sisa buangan dikurangkan. Bagi setiap masalah pengoptimunan, profil suhu yang optimum telah diperolehi adalah kurang daripada suhu optimum yang diperolehi sebelumnya. Kesimpulannya, keputusan pengoptimuman yang diperolehi dari profil suhu optimum OP3 ialah keadaan suhu yang terbaik untuk dilaksanakan dalam industri biodiesel kerana ia mampu menyediakan operasi yang lebih selamat dan menghasilkan pengeluaran biodiesel yang lebih besar dengan jumlah pengeluaran sisa yang lebih rendah dalam masa kelompok yang minimum.

SIMULATION AND OPTIMIZATION OF BIODIESEL PRODUCTION FROM DIMETHYL CARBONATE IN BATCH REACTOR

ABSTRACT

Simulation of biodiesel production from dimethyl carbonate (DMC) is very scarce and limited to utilization of canola oil as the raw material. In this study, a pilot scale of DMC transesterification process to produce biodiesel in a batch reactor was considered. A pilot scale operation of the batch reactor was introduced to the biodiesel production as a preparation for commercial and industrial development. A simulation of the pilot scale batch reactor for DMC transesterification reaction using Aspen Plus Version 2006 is presented in this study.

The simulation model was validated with experimental results available in the literature. The simulation model was validated at temperature of 75°C which gave $R^2 = 0.96$ and produced a similar trend of the Palm Methyl Ester (PME) concentration profile with the experimental results. From the validated simulation model, a pilot scale batch reactor operation was simulated and sensitivity studies were carried out to study the behavior of DMC transesterification process at a larger scale. A pilot scale batch reactor considered has a volume of 300 L with scaling factor of 660 from the experimental scale of operation. Based on the sensitivity studies, reactor temperature and amount of reactant were selected as variables to be optimized in the optimization study.

The optimization study was carried out with three objectives which were: maximize conversion (OP1), minimize batch time (OP2), and maximize palm methyl ester (PME) and minimize waste simultaneously (OP3). The optimization study was performed using the optimizer tool in Aspen Plus which was based on the sequential

quadratic programming (SQP) technique. The results showed that in optimization problem (OP)1, the conversion of DMC transesterification reaction increased from 90.6% to 99.9%. In OP2, the batch time required for the reaction was shortened from 8 hours to 2.68 hours while in OP3, the PME production significantly increased from 31.05 kg to 125.99 kg and the waste decreased from 76.42 kg to 63.78 kg at the shortest batch time specified.

In real situation, operational temperature of batch reactor is changing with time. Therefore, apart from optimizing temperature at single time interval, optimizing temperature at multiple time intervals was considered to obtain optimal temperature profile. By optimizing the profile, the performance of DMC transesterification process produced better results. The PME production increased while the batch time was shortened and with low amounts of waste produced. For each optimization problem, the optimum temperature profile obtained was less than the optimum temperature obtained at single time interval. In conclusion, optimization results obtained from optimal temperature profile of OP3 is the best temperature condition to be implemented in biodiesel industry since it was able to provide safer operation and produce larger biodiesel production with lower amount of waste produced at minimum batch time.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Energy sources can be classified into three groups namely fossil, fissile and renewable energy. Fossil fuels, for example petroleum, natural gas, coal, oil shale, bitumen and tar sands refer to an earlier geological age and are not renewable. The term 'fissile' is applied to materials that are fissionable by neutrons with zero kinetic energy. The main sources of fissile energy are uranium and thorium. Uranium is a scarce resource and is expected to last only for the next 30 to 60 years (Pimentel *et al.*, 1997). Moreover, the biggest challenge in nuclear plant is to find the safest way for waste disposal. Other than that, this plant has high operating and maintenance costs. Therefore, the application of fissile energy can even cause a worse scenario due to emission of harmful radiation. Among all the three types of energy, renewable energy is the best alternative because of its potential to provide an energy source with zero or almost zero emission of both air pollution and greenhouse gases.

One of the most favourite renewable resource is biodiesel because it contains very little sulphur, polycyclic aromatic hydrocarbons, and metals (Meher *et al.*, 2006). The fact that biodiesel does not contain polycyclic aromatic hydrocarbons makes it a safe alternative for storage and transportation. Since biodiesel is oxygenated, it is a better lubricant than diesel fuel, increases the life of engines, and is combusted more completely. The higher flash point of biodiesel makes it a safer fuel to use, handle, and store. With its relatively low emission profile, it is an ideal fuel for use in sensitive environments especially in heavily polluted cities (Pereira *et* *al.*, 2014). The most popular biodiesel is known as methyl ester, which is commonly produced from vegetable oil like soybean oil, palm oil, rapeseed oil, sunflower oil and safflower seed oil (Singh, 2010). In Malaysia, more attention is paid on the utilization of palm oil because Malaysia is the second largest producer of palm oil in the world. However, the use of palm oil in biodiesel production becomes a competitor to the oleochemical industry and food processing industry where palm oil is also a major feedstock (Johari *et al.*, 2015).

Due to the competition, a feasibility study of biodiesel production using palm oil is carried out. The selection of the best biodiesel process in consideration of the operational cost, environmental impact and safety issues may encourage a healthy biodiesel industry in the future. Based on previous studies, there are two basic methods used to convert palm oil into biodiesel, which are transesterification with alcohol and catalytic cracking of palm oil to lower molecular hydrocarbon (Sivasamy *et al.*, 2009). In general, most of the biodiesel plants in the world produce biodiesel using the transesterification reaction because it is the most economical process which requires low temperature and pressure with higher conversion yield. Besides that, the reaction is simple where palm oil reacts with alcohol (methanol/ ethanol/ propanol/ butanol) in the presence of a catalyst (Meher *et al.*, 2014).

Conventionally, palm oil will react with methanol in the existence of alkali or acid catalyst such as potassium hydroxide (KOH), sodium hydroxide (NaOH) and sulphuric acid (H_2SO_4) in the homogeneous condition. However, the homogeneous condition is not preferable because it has to be separated from the byproduct for further use. Besides, the treatment of alkaline wastewater after the reaction is difficult, environmentally unfriendly and requires much energy. In order to avoid the problems mentioned above, a heterogeneous type of process is preferable. In fact, a heterogeneous catalyst can minimize environmental impact, incur 4-20% lowering in refining cost and can simplify the purification process and make the expected operational cost be effectively low-cost (Zhang *et al.*, 2010a). Therefore, a heterogeneous condition could be the best method to be implemented in the transesterification reaction.

Dimethyl carbonate (DMC) transesterification reaction is a new transesterification process as an alternative method to biodiesel production using heterogeneous condition. In this reaction, the byproduct produce is more valuable, which is glycerol carbonate (GDC) as compared with abundant availability of glycerol from conventional method (Ilham and Saka, 2009). Besides that, DMC has high solubility with palm oil, thus extra solvent like tetrahydrofuran is not required. DMC can act as a solvent which can enhance the mass transfer in transesterification reaction. Excess amounts of DMC can easily shift the reaction equilibrium towards the formation of biodiesel (Zhang *et al.*, 2010a). Apart from that, this reaction can accommodate a safer chemical plant by having a DMC storage tank rather than a methanol storage tank. Chemically, methanol is a flammable and toxic liquid whereas DMC is non-toxic, non-irritating, biodegradable, stable and easy to handle.

Biodiesel production using DMC transesterification reaction contains more oxygen atoms due to unique structure of DMC. The physical properties of the biodiesel produced are not the same as that produced from methanol. The biodiesel from DMC will have excessive oxygen atoms and is reported to have a high flash point which is useful for fuel combustion. Moreover, the formation of fatty glycerol carbonates (FAGCs) as the byproduct in DMC transesterification reaction also plays a role in fuel combustion but there is some detrimental effect on flow properties (Fabbri *et al.*, 2007). Other biodiesel properties like acid number, kinematic viscosity, density and water content are within American Society for Testing and Materials (ASTM) specification of fuel (Dawodu *et al.*, 2014). Those properties make biodiesel production from DMC transesterification reaction potentially beneficial in the substitution of diesel fuel.

Due to positive feedback of biodiesel in diesel fuel, the demand is growth and hence, reveals the potential of DMC transesterification reaction at a larger scale of operation. A simulation study was practical in representing the real process. It can be developed using a computer simulation software such as Aspen Plus and Hysys software. In developing simulation models for chemical process, Aspen Plus and Hysys software are commonly used due to the availability of thermodynamic packages, vast component library and advanced calculation techniques (Morais *et al.*, 2010). Between Aspen Plus and Hysys software, Aspen Plus is more convenience and user-friendly. Besides that, Kurle *et al.*, 2013 has presented a rigorous simulation of DMC transesterification process using Aspen Plus.

Optimization studies in biodiesel production has received great attention. Some of the studies have conducted optimization to achieve operations at minimum cost, minimum environmental impact and minimum energy consumption. In achieving the target, the process has to be operated at optimum operation conditions or simplified to a simple transesterification unit operation (Kaewcharoensombat *et al.*, 2011).

Besides that, the optimization of the unit operation such as the batch reactor has been introduced (Denbigh, 1958). Batch reactor operation requires a long reaction time and the operation is operated on a small scale. The performance of the batch reactor is improved by changing the reactor temperature at several time intervals. Changing the reactor temperature at the optimal value is able to maximize conversion of the reactant and minimize batch time. At certain number of time intervals design, the quality and quantity of the desired product is achieved and the batch reactor can be efficiently and economically operated (Aziz and Mujtaba, 2002).

1.2 Problem Statement

Biodiesel via DMC transesterification reaction is very promising alternative to the conventional methanol transesterification reaction due to the nature properties of DMC and solvent-free reaction which can lead to safer and greener production of biodiesel. However, the studies available for this reaction were limited at laboratory scale only where the scopes of studies cover the reaction mechanism, reaction kinetic and reaction condition from various types of raw materials and catalyst (Su *et al.*, 2007; Tian *et al.*, 2009; and etc.). Moreover, studies on modelling and simulation of the DMC transesterification process are very scarce.

Process modelling and simulation is required for continuous improvement of chemical process with minimal time and cost especially for large scale of operation. In fact, the number of experiment during scale-up or even during commissioning of chemical plant can be reduced if reliable modelthat able to represent the real process can be developed. In this new era of globalization, technologies become more sophisticated. Computer-aided technologies such as Aspen Plus and Hysys software are very useful in process modelling and simulation and has been well accepted in chemical processes and industry.

According to Zhang et al. (2010a), DMC transesterification reaction of palm oil was carried out using batch reactor. The batch operation required 8 hours of reaction time to produce 96.2% of biodiesel production. The reaction time is longer and hence, hindered the production of biodiesel to be implemented at the industrial scale. To overcome that problem, process optimization of batch DMC transesterification reaction of palm oil is indispensable to maximize biodiesel production or to minimize batch time, which can lead to the optimum operation at a more feasible time. Moreover, biodiesel industry with minimize waste production is expected to perform well in competing and restricting regulation of biodiesel industry. Due to those conditions, the optimum operation to achieve such objectives is important and needs to be considered before the operation can be implemented in real industrial scale.

1.3 Research Objectives

The objectives of this study are:

i. To develop simulation model of DMC transesterification reaction.
ii. To perform sensitivity analysis to study the behavior of DMC transesterification reaction.
iii. To optimize the batch operation of DMC transesterification reaction.

1.4 Organization of the thesis

This thesis is organized into five chapters.

In Chapter 1, the general idea of the current work is presented in the research background. Next, the problem statement reveals the related issue regarding the research area and the explanation of a possible solution is discussed. In addition, the purpose of this research is also highlighted via the research objectives. Finally, a summary of the chapter is provided at the end of this chapter.

In Chapter 2, a brief review of the biodiesel production and the chemical process involved is given at the beginning of this chapter. Then, the DMC transesterification reaction, which is the main process considered in this study, is explained further. A literature review on the plant capacity of the transesterification reaction, the simulation of the biodiesel process and the optimization of the biodiesel process is also presented. Then, the flexibility of the batch reactor in biodiesel production is also reviewed and finally, the strategy to optimize the batch reactor from different approaches is provided.

In Chapter 3, the methodology used in this research is explained. At the beginning, the steps involved for the simulation model of the biodiesel process in Aspen Plus software are explained. Then, the standard methodology for the scale up process is defined. Next, the procedures for the sensitivity analysis study for the biodiesel process are elaborated and finally, the methods to perform the optimization study for the biodiesel process are clearly shown in this chapter.

In Chapter 4, the research results are presented and discussed. The results presented include the validation of the simulation model of DMC transesterification process, the pilot scale up of DMC transesterification reaction, and the sensitivity analysis studies. Besides that, the optimization studies results with three optimization problems i.e. maximize conversion, minimize time and maximize PME and minimize waste simultaneously are discussed. Each of the results obtained is further discussed and compared.

In Chapter 5, all the important findings in this study are summarized and some suggestions for future study are also proposed.