A COMPARATIVE STUDY ON THE JET LOOP REACTOR AND CONTINUOS STIRRED TANK REACTOR IN THE SELECTIVE HYDROGENATION OF PALM OLEIN (I.V.64)

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A thesis submitted in fulfilment of the requirements for the award of the degree of Master of Engineering (Chemical)

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MAY 2005

Dedicated to My Beloved Family and Friends

ACKNOWLEDGEMENT

Firstly, I would like to express my sincere gratitude to my supervisor, Associate Professor Dr. Rosli Mohd Yunus of Chemical Engineering Department, for his dedication, support and guidance throughout the whole period of this research work. His knowledge and experience in the field of hydrogenation process has enlightened me to be involved in this relatively new area of Jet Loop Reactor (JLR). I also appreciate his guidance on the research and the freedom that he had given me in exploring the scopes for my research. His encouragement and knowledge support since my final year research project during the undergraduate study is very much appreciated.

I am also very grateful to SOCTEK Edible Oil Sdn. Bhd. for supporting me in the research. Special thank to the Vice President (Manufacturing/Technical), Mrs.Gowrie Jayaraman who has granted me her support in doing the lab scale Continuous Stirred-Tank Reactor (CSTR) in the plant premises. Her kindness in sponsoring me the raw materials needed for the hydrogenation process, such as palm olein I.V. 64 and High trans catalyst, and the permission given to use all the testing facilities in order to check the hydrogenated product from both the JLR and CSTR systems, is indescribably thankful. I am also grateful to her fellow staffs, Mr. Teo Kok Leong and Mr Azahari who have given me full support by giving me relevant comments and ideas regarding the research.

I am grateful to Universiti Teknologi Malaysia for providing me with the UTM-PTP scholarship.

Finally, I would like to thank my parents, siblings and Liew Yew Loung for their support and understanding during my difficulties.

ABSTRACT

Jet Loop Reactor (JLR) was developed to improve the overall performance of hydrogenation processes. Nevertheless, the application of JLR in the palm oil and oleochemical industries in Malaysia is still very much sparse. A substantial amount of investment and the lack of study conducted in Malaysia on the application of JLR have retarded the retrofitment and/or replacement of the conventional CSTR with this technology. In the wake of this, a comparative study was conducted to investigate the performance of JLR in the selective hydrogenation of palm olein with an IV of 64 in comparison to the hydrogenation in the conventional CSTR system. A pilot scale JLR with a capacity of 250 liter was used in the study. The circulation of the sample in the loop was achieved via a single speed pump. The experimental result was compared with result from the CSTR experiment. A down-scaled laboratory CSTR apparatus was used in the study for this purpose. A software package, developed via Microsoft Excel 2000 and Visual Basic Application (VBA) softwares, was used to simulate the behavior of the hydrogenation process in both, JLR and CSTR, under similar capacity. The outcome of the study showed that with the limitation of single speed pump, the JLR could not matched the superiority of CSTR in the selective hydrogenation process for it required slow reaction to produce high trans fatty acids hydrogenated product. On the positive note, the developed software package is a useful tool which allows an easy method to study the behavior of the hydrogenation process of JLR and CSTR. The prediction of the CSTR process was acceptable, but the prediction of JLR process was less accurate, revealing a maximum of 30% error. It can be concluded that the present analytical method used in the simulation of JLR required improvement on the modeling of the process, or to opt for the numerical solution, to produce a much better prediction. A retrofit method was also suggested in the study, for the possibility of fitting in the JLR facility in the existing CSTR system with minimal modification, for the system to have dual function of slow and fast reactions.

ABSTRAK

Reaktor Jet Loop (JLR) dibangunkan untuk memperbaiki kecekapan keseluruhan proses penghidrogenan. Walaubagaimanapun, JLR amat jarang digunakan di dalam industri minyak kelapa sawit dan oleokimia di Malaysia. Faktor pelaburan yang agak tinggi serta kekurangan penyelidikan yang telah dijalankan di Malaysia, telah membantutkan proses mengubahsuai dan/atau mengganti sistem konvensional reaktor pengacauan berterusan, CSTR, dengan teknologi yang telah dibangunkan ini. Atas alasan ini, suatu kajian perbandingan telah dijalankan untuk mengkaji kecekapan sistem JLR di dalam proses penghidrogenan terpilih minyak sawit olein dengan nilai iodin (IV) 64, berbanding penghidrogenan menggunakan sistem CSTR. Suatu sistem JLR berskala loji pandu dengan kapasiti 250 liter, telah digunakan bagi tujuan ini. Pengitaran sampel di dalam sistem JLR ini dihasilkan oleh suatu pam yang mempunyai satu kelajuan. Keputusan ujikaji ini dibandingkan dengan keputusan kajian yang dijalankan dengan menggunakan sistem CSTR. Suatu peralatan CSTR berskala makmal yang direkabentuk berdasarkan peralatan CSTR di industri, telah digunakan bagi tujuan ini. Suatu pakej perisian telah dibangunkan, menggunakan perisian Microsoft Excel 2000 dan Visual Basic Application (VBA), bagi tujuan penyelakuan proses penghidrogenan di dalam sistem JLR dan CSTR, di dalam kapasiti yang sama. Hasil dari kajian ini menunjukkan bahawa, dengan penggunaan pam satu kelajuan, sistem JLR tidak dapat menandingi kehebatan sistem CSTR di dalam penghidrogenan terpilih kerana penghidrogenan itu memerlukan proses tindakbalas yang perlahan untuk menghasilkan produk dengan kuantiti asid lemak *trans* yang tinggi. Walaubagaimanapun, pakej perisian yang telah dibangunkan merupakan suatu kemudahan yang berguna yang menyediakan suatu kaedah mudah di dalam mengkaji kelakuan proses penghidrogenan di dalam sistem JLR dan CSTR. Ramalan terhadap proses CSTR adalah baik. Tetapi ramalan terhadap proses JLR memberikan ramalan yang sedikit tersasar, dengan ralat maksima sebanyak 30%. Adalah disimpulkan bahawa kaedah analitik yang digunakan untuk menyelesaikan penyelakuan sistem JLR perlu pembaikan di dalam model yang digunakan di dalam penyelakuan ini, atau menggunakan kaedah berangka di dalam menyelesaikan penyelakuan tersebut, supaya dapat menghasilkan ramalan yang lebih baik. Suatu kaedah pengubahsuaian juga dicadangkan di dalam kajian ini bagi memasukkan kemudahan JLR ke dalam sistem CSTR sedia ada di industri bagi membolehkan sistem tersebut mempunyai dua fungsi, untuk menjalankan proses penghidrogenan dengan tindakbalas cepat dan tindakbalas perlahan.

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- Specific mass transfer area (m^2/m^3) а Concentration (mol/m³) С _ Initial concentration (mol/m³) Ci _ Input concentration (mol/m³) Co d Diameter (m) _ Gas flow (m^3/s) G _ Η Reactor height (m) k Mass transfer coefficient (m/min) _ K Rate Constant (1/min) _ l Length (m) _ Liquid flow (m³/s) L _ L Lower part Mass (kg) m -Amount of substance (mol) п -Molar flow (mol/min) 'n _ Retardation factor R _ S Solubility of hydrogen in oil (vol/vol) _ Temperature (°C) t Time (min) t Duration of solute pulse (min) t_o Т Reactor diameter (m) $v \sqrt{\left(1 + \frac{4\mu D}{v^2}\right)}$ u Upper part U Velocity (m/s) v _ Volume (m^3) V_
- \dot{V} Volumetric flow (m³/min)

W	-	Velocity (m/s)
x	-	Length of loop (m)

Greek Letters

γ	-	General zero-order rate coefficient for production
\in_L	-	Liquid holdup
τ	-	space time ($\tau_2 = V_L / \dot{V}_{circ}$)
μ	-	Viscosity (kg/ms)
μ	-	General first-order rate coefficient for decay
ρ	-	Catalyst bulk density (kg/m ³)

Superscripts and Subscripts

cat	-	Catalytic
circ	-	Circulation
ej	-	Ejector
g	-	Gas phase
i	-	Component index
k	-	Reactor index
1	-	Liquid phase
Μ	-	Active metal
n	-	Nozzle
nonc	at	- Non-catalytic
0	-	Feed
t	-	Throat
,	-	Reaction vessel
,,	-	Loop
,,,	-	Ejector

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Hydrogenation process is widely used commercially to increase the melting point and to improve the consistency of oils and fats. Hydrogenation also reduces colour and odour, improves thermal stability and resistance to oxidation of fats and oils (Yap *et al.*, 1989; Busfield *et al.*, 1990; Smidovnik *et al.*, 1992; Choo *et al.*, 2001; Karabulut *et al.*, 2003). In the hydrogenation process, part of double bonds are eliminated while a significant proportion of the remaining bonds are isomerized through cis/trans conversion on positional shifted in the fatty acid chain. Herein, the isomerization contribute to the selectivity of a hydrogenation process. A fatty acid chain with higher selectivity is claimed to have higher trans isomers compare to the cis isomers (Swern *et al.*, 1979; Jovanovic et al., 1998; Karabulut *et al.*, 2003). Composition and properties of the final product depend on various operating factors, including catalyst type and concentration, agitation, hydrogen pressure and temperature (Busfield *et al.*, 1990; Jovanovic et al., 1998; Choo *et al.*, 2001; Salmi *et al.*, 2002; Krabulut *et al.*, 2003).

Various means exist to create the physical conditions in bringing together oil, hydrogen and catalyst, namely, circulation system, dead-end system and both circulation and dead-end system. Using the above mentioned systems, hydrogenation is done either in batch or continuous process. However, due to the variation in raw materials and desired end products, application of continuous hydrogenation remains limited; therefore, most hydrogenation is done in batch autoclaves (Patterson, 1983; van Dierendonck *et al.*, 1998). A batch autoclave or a conventional Continuous Stirred-Tank Reactor (CSTR) is commonly utilized in the hydrogenation process. It is also one of the most commonly used devices in industry for mixing (Yoon *et al.*, 2001). Nevertheless, proper design of turbine-stirred-tank reactor on an industrial scale can still be difficult to make (Dohi *et al.*, 2002; Yoon *et al.*, 2001). On the large scale, the removal of heat may become a limiting factor. Installation of additional cooling coils into the reactor vessel makes the design problems even more complex (van Dierendonck *et al.*, 1998).

Therefore, a Jet Loop Reactor (JLR) is claimed to retrofit well the CSTR and represent a very attractive alternative technology for hydrogenation process. Due to the increasing demands of effective hydrogenation process system, nowadays, many researchers have involved themselves in many projects to study the feasibility of alternative Jet Loop Reactor (JLR) to replace the present conventional Continuous Stirred-Tank Reactor (CSTR) in their systems (Havelka *et al.*, 1997; Van Dierendonck *et al.*, 1998; Stefoglo *et al.*, 1999; Cramers *et al.*, 2001; Broekhuis *et al.*, 2001). A typical Jet Loop Reactor (JLR) consists of a vessel, an ejector and a circulation loop equipped with a pump. The benefit of this reactor is its efficiency in gas-liquid mass transfer, which is accomplished by means of the ejector. Typically, no mechanical agitation is required, and heat transfer problem is solved by using an external heat exchanger (Van Dierendonck *et al.*, 1998; Lehtonen et al., 1999; Broekhuis *et al.*, 2001). Hence, undesired problem areas are solved and series of advantageous are offered to the users.

1.2 Objectives

The main purpose of this research was to study the feasibility of retrofitting the conventional Jet Loop Reactor (JLR) with Continuous Stirred-Tank Reactor (CSTR)

system by performing a comparative study on Jet Loop Reactor (JLR) and Continuous Stirred-Tank Reactor (CSTR) in the selective hydrogenation of palm olein I.V. of 64.

In the selective hydrogenation, it is aimed to reach to a certain iodine number and also polyunsaturated acids are converted to monounsaturated acids (Karabulut *et al.*, 2003). Herein, an Iodine Value (I.V.) drop of 10 is aimed in the study. Besides that, a selective hydrogenation which requires less plentiful of hydrogen is chosen as the critical comparison in the research. The selective hydrogenation is commonly well performed using Continuous Stirred-Tank Reactor (CSTR) as it contributes a less plentiful of hydrogen. Jet Loop Reactor (JLR), on the other hand, is well suited for a non-selective hydrogenation (fewer mass transfer limitation).

Hence, it is the objective of the research to study whether the Jet Loop Reactor (JLR) is suitable for both selective and non-selective hydrogenation. Both the Jet Loop Reactor (JLR) and Continuous Stirred-Tank Reactor (CSTR) used in the research were presented by a pilot plant system with a maximum capacity of 250 litres for JLR and a full laboratory system with a maximum capacity of 1.5 litres for CSTR. Both systems used here were the down scaled version of the industrial scale system. Same type of operating conditions and raw materials were used in the hydrogenation process. The systems were scaled to a comparative capacity before the results were analyzed in the research study.

1.3 Scopes of Study

In order to achieve the objective of the study, the following research steps were taken. The research consisted of several important parts. Summary of the research scope was shown in Figure 1.1. The mentioned parts involved were:



Figure 1.1: Research methodology flow chart

1.3.1 Experimental Study on Lab Scale Hydrogenation Process

Experiments were conducted using palm olein as raw material of lab scale CSTR hydrogenation process. Data were collected from the system and analyses were done on the acquired data.

1.3.2 Experimental Study on Pilot Scale Hydrogenation Process

Experiments were conducted using palm olein as raw material of pilot scale JLR hydrogenation process. Data were collected from the system and analyses were done on the acquired data.

1.3.3 Conventional Programming, Modeling and Simulation of JLR and CSTR

The data received from the experiments were used as the default values of modeling and simulation. A mathematical modeling of Jet Loop Reactor (JLR) was developed followed by numerical solution of the resultant model. Both mathematical and numerical solutions were applied in the conventional programming software, Microsoft Excel and Visual Basic Application to model the real system of Jet Loop Reactor (JLR). The resultant system was verified using data collected from the experimental study.

1.3.4 Analyses and Comparative Study of JLR and CSTR

Both systems were analyzed and compared. Discussions were made on the ability to retrofit the Jet Loop Reactor (JLR) in place of the Continuous Stirred-Tank

Reactor (CSTR) by means of experimental study and validity test using Jet Loop Reactor (JLR) simulation and modeling. Suggestions for further improvement in the future were done after the conclusions of the research were made.

1.4 Research Overview

According to the scopes of the study, the research was divided into two major parts:

- (a) Data Collection and Parameters determination.
- (b) Computer modeling and simulation.

1.4.1 Data Collection and Parameters Determination

Two sets of experiments were done in this project in order to collect required data and were used to determine the parameters required. For Continuous Stirred-Tank Reactor (CSTR), a set of experiments using lab scale equipment was done in SOCTEK (M) Edible Oil Sdn. Bhd. Data and parameters influencing the hydrogenation of palm olein using Continuous Stirred-Tank Reactor (CSTR) were collected and identified. Similar experiments were done with the same parameters but using Jet Loop Reactor (JLR). Same type of raw material such as palm olein, nickel catalyst and operating conditions were utilized in this project.

1.4.2 Computer Modeling and Simulation

A mathematical modeling was done on Jet Loop Reactor (JLR). The JLR was divided into three essential parts, namely reaction vessel, ejector and loop part. Each part of the JLR was modeled using gas and liquid mass balances. Tanks in series and dynamic axial dispersion model was used to model JLR. Analytical methods algorithm were used to solve the mathematical model.

Conventional programming language, Visual Basic Application together with Microsoft Excel was used to present the data obtained from modeling within spreadsheet environment. The model obtained was further verified with experimental results. Further on, the model developed were used to develop as similar as possible to the conventional simulator being used.

1.5 Importance of the Study

A few contributions and importance of the study were notified from the study, namely:

- a) To give a general view of the pilot plant hydrogenation of palm olein
 I.V. 64.
- b) To give information on the possibility of retrofitting the CSTR with JLR.
- c) To introduce new software, this can be utilized as a modeling and simulation program of CSTR and JLR.

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