PREPARATION OF SULFONATED INCOMPLETE CARBONIZED GLUCOSE ACID CATALYST VIA MICROWAVE IRRADIATION FOR MEHTYL ESTER PRODUCTION

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ABSTRACT

The usage of homogeneous catalyst in esterification process of feedstock with high free fatty acid (FFA) resulted in higher cost for post-production, wastewater treatment, and also catalysts loss. This gives reason for development of catalyst to substitute sulphuric acid in the industry. Conventional heating method of heterogeneous acid catalyst seems promising however, the heating time of carbonization and sulfonation always take longer and is not economically viable. Catalysts prepared by conventional heating also have low surface area and acid site density. Microwave-assisted sulfonated glucose was proposed in this study with several modifications of its parameter to fit in the industry scale. Microwave-assisted heating method initiates the rate of reaction and shortens the time and energy required using the power of radiation. A catalyst made from renewable resources, D(+)-Glucose was carbonized based on a few screening processes and the incomplete carbonized glucose (ICG) was then sulfonated with H_2SO_4 (SO_3H/ICG). SO_3H/ICG was characterized by using elemental analysis, X-ray diffraction, temperature programmed desorption of ammonia, field emission scanning electron microscope, Brunauer-Emmett-Teller, Fourier transform infrared spectroscopy as well as the dielectric constant (ε'), loss factor (ε'') and tangent loss ($\tan \delta$). D catalyst which was prepared at 20 min heating time, 20g D(+)-Glucose using 400W microwave power level showed remarkable surface area of 16.94 ± 0.30 m^2/g, highest acid site density of 25.65 mmol/g and lowest ε', ε'', $\tan \delta$ of 11.275, 10.691 and 0.948 respectively. Then, the catalyst was tested in esterification process of palm fatty acid distillate (PFAD) and analysis of the PFAD biodiesel was done through gas chromatography - flame ionization detector. The best performing catalyst, $SO_3H/ICG_{(7)}$ with 91.41 % yield and 90.93 % conversion was then optimized via response surface methodology in order to obtain the optimum operating condition for sulfonation process. Through optimization, the most optimum reaction time, H_2SO_4 volume, power level of microwave and stirring rate were found to be 7.53 minutes, 159.51 ml, 413.64W and 670.53 rpm in order to obtain highest percentage yield of 94.01% and percentage conversion of 91.89%. Kinetic study was developed throughout the esterification process and activation energy from the forward and reverse reaction were found to be 3.36 kJ/mol and 11.96 kJ/mol respectively. The cost of biodiesel production from PFAD using microwave heating system on the other hand was higher for laboratory scale which was RM 31.33/kg compared to pilot and industrial scale of RM 3.29/kg and RM 1.76/kg, respectively. However these costs were much cheaper compared to conventional cost which reached RM 57.57/kg for laboratory scale. In conclusion, esterification process of PFAD to biodiesel using sulfonated glucose prepared via microwave-assisted heating method offers a greener procedure for catalyst preparation which is more convenient in time and cost saving.

ABSTRAK

Penggunaan mangkin homogen dalam proses esterifikasi bahan yang mempunyai asid lemak bebas (FFA) yang tinggi memerlukan kos yang lebih tinggi untuk pasca pengeluaran, rawatan air sisa, dan juga mangkin yang hilang. Inilah sebabnya mengapa perkembangan dalam mewujudkan mangkin pepejal yang bersifat heterogen berasid diperlukan untuk menggantikan asid sulfurik dalam industri. Kaedah pemanasan mangkin heterogen asid secara konvensional kelihatan bagus, namun proses karbonisasi dan sulfonasi selalu memakan masa lebih lama dan tidak dapat dilaksanakan secara ekonomi. Mangkin yang disediakan secara pemanasan konvensional juga mempunyai luas permukaan dan ketumpatan tapak asid yang rendah. Sulfonikasi D(+)-glukosa dengan bantuan gelombang mikro diusulkan dalam kajian ini dengan beberapa pengubahsuaian parameternya agar sesuai dengan skala industri. Kaedah pemanasan berbantukan gelombang mikro memulakan kadar tindak balas dan memendekkan masa dan tenaga yang diperlukan menggunakan radiasi. Mangkin yang dibuat dari sumber yang boleh diperbaharui, D(+)-Glukosa dikarbonisasi berdasarkan beberapa proses penyaringan dan glukosa berkarbonat yang tidak lengkap (ICG) kemudian disulfonasikan dengan H_2SO_4 (SO_3H / ICG). SO_3H / ICG dicirikan dengan menggunakan analisis unsur, pembelauan sinar-X, penyerapan suhu terprogram-ammonia, pengimbasan medan pelepasan mikroskop elektron, *Brunauer-Emmett-Teller*, spektroskopi inframerah transformasi Fourier serta pemalar dielektrik (ε'), faktor kehilangan (ε'') dan kehilangan tangen ($\tan \delta$). Mangkin D yang disediakan pada masa pemanasan 20 minit, 20g D(+)-glukosa dan menggunakan tahap kuasa gelombang mikro 400W menunjukkan luas permukaan yang luar biasa sebanyak 16.94 ± 0.30 m^2 / g, ketumpatan tapak asid tertinggi (25.65 mmol/g) dan nilai ε', ε'', tan δ yang rendah masing-masing adalah 11.275, 10.691 dan 0.948. Kemudian, mangkin diuji dalam proses esterifikasi sulingan asid lemak sawit (PFAD) dan analisis biodiesel PFAD dilakukan melalui kromatografi gas-pengesan pengionan api. Mangkin yang menunjukkan prestasi terbaik, $SO_3H/ICG_{(7)}$ dengan hasil 91.41% dan penukaran 90.93% kemudian dioptimumkan melalui kaedah gerakbalas permukaan untuk mendapatkan keadaan operasi optimum untuk proses sulfonasi. Melalui pengoptimuman, masa tindak balas yang paling optimum, isipadu H_2SO_4, tahap daya gelombang mikro dan kadar pengadukan masing-masing adalah 7.53 minit, 159.51 ml, 413.64W dan 670.53 rpm untuk memperoleh peratusan hasil 94.01% dan peratusan penukaran 91.89% yang tertinggi. Kajian kinetik telah dijalankan untuk proses esterifikasi ini dan tenaga pengaktifan dari tindak balas maju dan mundur masing-masing adalah 3.36 kJ/mol dan 11.96 kJ/mol. Kos pengeluaran biodiesel dari PFAD menggunakan sistem pemanasan gelombang mikro sebaliknya adalah lebih tinggi untuk skala makmal iaitu RM 31.33/kg berbanding skala pilot dan perindustrian masing-masing adalah RM 3.29/kg dan RM 1.76/kg. Walau bagaimanapun, kos ini jauh lebih murah berbanding dengan kos konvensional yang mencecah RM 57.57/kg untuk skala makmal. Kesimpulannya, proses esterifikasi PFAD ke biodiesel menggunakan glukosa sulfonasi yang disediakan melalui kaedah pemanasan berbantukan gelombang mikro menawarkan prosedur yang lebih hijau untuk penyediaan mangkin yang lebih mudah dalam menjimatkan masa dan kos.

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LIST OF ABBREVIATIONS

AA	-	Acyl Acceptor
ASTM	-	American Society for Testing and Materials
DOE	-	Design of Experiment
E_a	-	Activation Energy
et al	-	et alia (and others)
FAAE	-	Fatty Acid Alkyl Ester
FAME	-	Fatty Acid Methyl Ester
FFA	-	Free Fatty Acid
GCMS	-	Gas Chromatography Mass Spectrometer
min	-	minutes
MR	-	Molar ratio
MW	-	Microwave
GHG	-	Greenhouse gases
PFAD	-	Palm Fatty Acid Distillate
R	-	Universal molar gas constant (Arrhenius equation)
RSM	-	Response Surface Methodology
T	-	Temperature (^oC)
wt%	-	Weight percentage
X	-	Factor of parameter

LIST OF SYMBOLS

^oC	-	Degree celcius
CO_2	-	Carbon Dioxide
NO_x	-	Nitrous Oxide
SO_2	-	Sulfur Dioxide
β_o	-	Intercept coefficient
β_i	-	linear coefficient
β_{ii}	-	Quadratic coefficient
β_i	-	Constant coefficient
Y	-	Yield
C	-	Conversion
X_1	-	Time (min)
X_2	-	Volume H_2SO_4 (ml)
X_3	-	Power Level (W)
X_4	-	Stirring rate (rpm)

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Demand on crude petroleum is getting increased each year proportionally with the increment of human population and industries. It became a major component in transportation sector and world economy (Administration, 2020). Major energy consumption in Malaysia is natural gas with 43% shares followed by petroleum and other liquids with 37% shares as illustrated in Figure 1.1 (Administration, 2017). The consumption on petroleum is causing the depletion of petroleum resources. Automobile and energy-based industry will be the most affected since they are facing with the consequences of shutting down their business (Bharathiraja et al., 2014a). Apart of that, emission of greenhouse gases (GHG) caused by the combustion of petroleum based product raised environmental issues. Gases such as CO_2, HC, NO_x, and SO_x which are being emitted from petroleum combustion bring negative impact towards respiratory system, human skin as well as to the environment (Dey and Mehta, 2020).

Researchers continuously seek for other alternative in order to replace the fossil fuel. They came up with biodiesel which comprised of mono alkyl esters of long chain fatty acids produced through transesterification or esterification process of vegetable oil or animal fat with an alcohol (Reis and Cardoso, 2016). Biodiesel became one of the alternative due to several advantages factors which include biodegradable, non-toxic, renewable resources, and environmentally safe (Amin, 2019). Besides playing role as engine fuel for transportation, biodiesel are also used to generate power in backup systems where emission matters most (Barua et al., 2020), bio-surfactants (Sari et al., 2019) and biodegradable polyesters (Bilck et al., 2015).

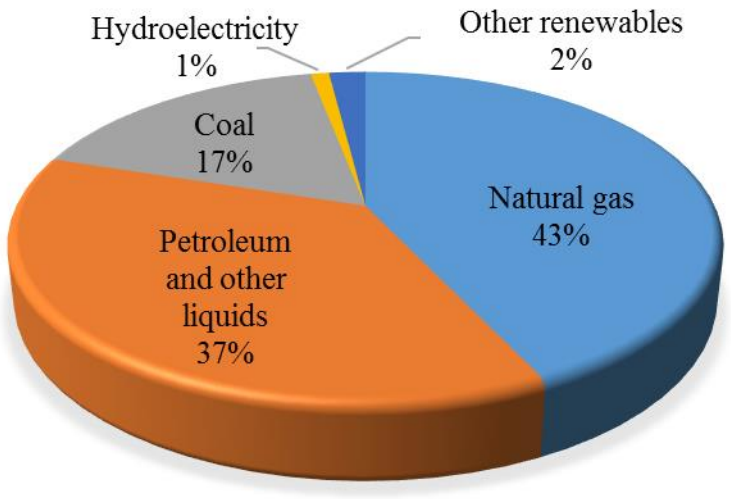

Figure 1.1 Malaysia's primary energy consumption, 2017 (U.S Energy Information Administration)

Biodiesel is a green fuel which is comparable to petroleum-based diesel. There are several major feedstock of biodiesel in Europe including soybean oil, rapeseed oil as well as canola oil and it can be easily found in United States and Canada. Some countries especially China, United States, Canada and Malaysia has been driven to utilized rendered animal fats and oils to enhance the biodiesel production (Adewale et al., 2015b). There were also research production on biodiesel productions using renewable feedstock such as algae oil (Khan et al., 2017), cotton seed (Sinha and Murugavelh, 2016), jatropha oil (Singh et al., 2021), karanja oil (Patel and Sankhavara, 2016) and many more which shows more than 95% of biodiesel production feedstock come from edible oils.

As for Malaysia context, palm oil is easily available nationwide. Palm oil is used along with alcohol for biodiesel synthesis. Palm is an ornamental tree. Around 95% edible oil and 5% non-edible waste oil are acquired during the extraction of palm oil from palm oilseeds. The by-product generated from refinery stage of palm oil process is known as palm fatty acid distillate (PFAD). It is brownish in colour and appears to be solid at room temperature. PFAD is non-edible due to high free fatty acid (FFA) content up to more than 80% (Ganesan et al., 2020). Aside from the stable properties, PFAD can be purchased at a cheaper price compared to any other

feedstock (Abdul Kapor et al., 2017). PFAD's price has fluctuated throughout the time. Figure 1.2 illustrates the price of PFAD since the past two decades. It is observed that the price of the PFAD reached its highest price in 2010 which was $992.5 FOB/Tonne. However, most of the time, the price of PFAD is low and it has been reduced to as low as $320 USD-FOB/Tonne in 2018. Since the price is low and the properties are consistent throughout the time, PFAD have been regarded as a remarkable low cost potential feedstock for biodiesel production.

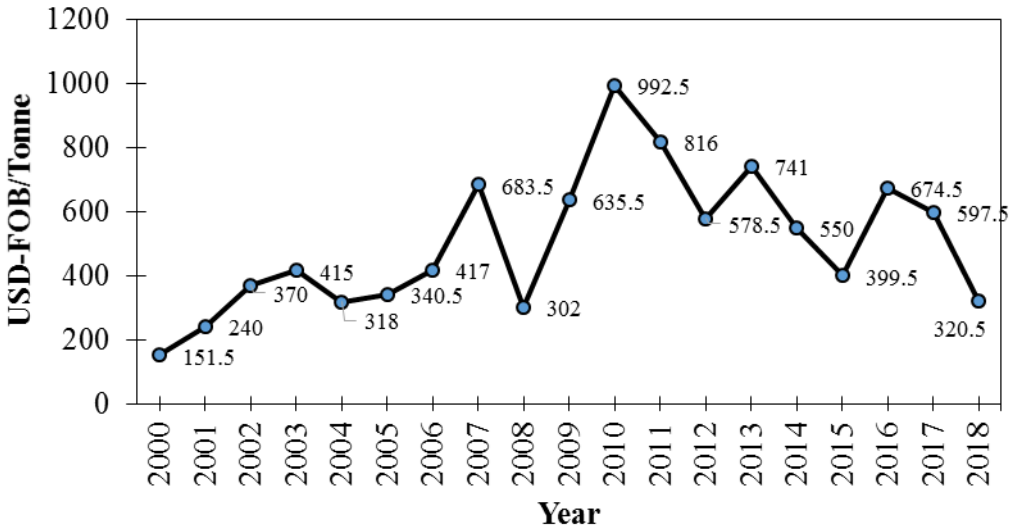

Figure 1.2 Price of PFAD-FOB/Tonne from 2000 until 2018 (Analytics, 2019).

There are many researches investigating on the development of catalyst towards esterification of PFAD. Since PFAD has high number of FFA, catalyst with high acidity is required to esterify FFA and transesterify the respective triglycerides simultaneously (Chaveanghong et al., 2018). There are two types of acid catalysts which are homogeneous acid catalyst and heterogeneous acid catalysts. Homogeneous acid catalyst is basically in the same phase with the reactant meanwhile heterogeneous acid catalyst is in different phase with the reactant. By using common homogeneous acid such as sulphuric acid (H_2SO_4), further post-treatment of the product is required in order to recover the product from the H_2SO_4 and this step is troublesome.

Application of a heterogeneous solid acid catalyst, in the other hand, could eliminate the unnecessary H_2SO_4 separation step in biodiesel production. It could potentially save time and the process is more environmentally friendly. Owing to this fact, heterogeneous solid acid catalyst is the most appropriate for biodiesel production from PFAD.

The most recent heterogeneous acid catalyst was developed from carbon-based material. Carbon-based such as D(+)-Glucose can be bought cheaply and it is made from numerous renewable resources such as oil palm frond juice (Zahari et al., 2012). It have been used as heterogeneous acid catalyst previously, however, the catalyst was produced conventionally (Lokman et al., 2015b) and this consumes longer time and higher temperature. Lokman managed to carbonized D(+)-glucose at 400 °C for 12 hours and sulfonated the incomplete carbonized glucose (ICG) at 150 °C for 15 hours long. Conventional heating method is the most adopted technique in past studies because of smooth heating process due to the gradual transfer of heat on the object's surface to the interior. However, in actual, the heating process from outside is slow, restricted to the highest temperature and caused inevitable deviations of temperatures (Cheng et al., 2013).

Microwave on the other hand is much more effective because the system is using thermal radiation with sensational penetrability (Xiao et al., 2011). As a matter of fact, microwave radiation stimulate the migration of ion and molecular motions of dipole rotation of alcohol, catalyst as well as fatty oils without altering their molecular structure (Ning and Niu, 2017a). Microwave radiation is proven to shorten the heating time and improve the heating rate. Liu et al. manages to synthesis fatty acid ethyl esters (FAEE) using aminophosphonic acid resin (D418) via MW and they shorten the reaction time from 12 h to 7 h (Liu et al., 2013b). Refaat et al. on the other hand successfully transesterified waste cooking oil (WCO) with methanol from conventional heating method to microwave assisted heating method and shortened the heating time from 60 min to 2 min (Refaat et al., 2008). Previous studies only investigated the length of time taken for respective work employing microwave (with fixed power) and not covering the overall specification on the microwave such as wave variation strength.

1.2 Problem Statement

Palm oil has been used widely in industry for biodiesel production, however in this study palm fatty acid distillate (PFAD) has been selected to be the feedstock due to its cheaper price and stable properties. However, esterification of PFAD requires a very highly acidic catalyst. In esterification, homogeneous acid such as sulphuric acid (H_2SO_4) is widely used. However, sulphuric acid is well known as an extremely corrosive chemical and triggers difficulties and environmental concern during its recovery process. That is why D(+)-Glucose , as a heterogeneous catalyst is proposed to overcome this problem. In fact, D(+)-Glucose is cheaper than sulphuric acid and has great potential and good performance towards esterification of PFAD. In the current development of acid catalyst, D(+)-Glucose is commonly sulfonated with H_2SO_4 and yield an average of 92% biodiesel. The catalyst prepared conventionally is heated through a complicated process where high temperature is employs in order to oxidizing a portion of mass or rendering it friable. In fact, the conventional heating method provide a catalyst with lower acid site density, surface area and conversion of FFA as well. The calcination process sometimes took hours some even days using a high temperature to remove the moisture and volatile impurities completely. Should this method be commercialized, higher cost and longer duration to produce sulfonated D(+)-Glucose catalyst will incur and would probably be not economically viable. The conventional heating method can be simplified via microwave irradiation where the chemical reaction can be accelerated, improve the quality of the prepared catalyst as well as reduce the consumption of time and energy. The microwave heating mechanism is a volumetric process in which heat is generated within the material itself, and, consequently, it can be very rapid and selective. In this way, the catalyst heated can absorb the energy embodied in the microwaves produces a high quality of surface area and acid site density thus capable to convert the FFA with a high value percentage.

1.3 Hypothesis

Common development of solid acid catalyst requires calcination and sulfonation which basically applies high temperature and time consuming. Thus, improvement of conventional method is required to directly reduce the time and temperature of the catalyst synthesis, which could be achieve from microwave technology. Microwaves work in conjunction with the electromagnetic waves with about 300 MHz and 300 GHz of frequencies. Instead of conventional heating, microwave heating is more efficient in terms of the speed and uniformity of heating process (Bhattacharya and Basak, 2016). Besides that, microwave heating direction is reversal compared to conventional heating. The microwave heating occurs from the core as microwave penetrates and generates heat inside the sample. The dielectric properties of materials are closely related with microwave frequency and temperature. Therefore, it is necessary to study dielectric properties of materials when adopting a microwave strategy. Consequently, this research was conducted to apply the microwave heating as an alternative to the conventional heating in order to rapidly reduce time, temperature, energy and cost.

1.4 Objectives of Research

i. To screen the carbonization process for D(+)-Glucose using microwave irradiation to obtain optimum parameters.
ii. To characterize and esterify the catalyst prepared based on carbonization parameters using fixed sulfonation process parameters and fixed esterification process parameters.
iii. To determine the dielectric properties of the catalyst prepared via microwave irradiation
iv. To optimize sulfonation process for incomplete carbonized glucose (ICG) using the best performing catalyst from carbonization screening via response surface methodology (RSM).
v. To establish reaction kinetics of the esterification reaction

1.5 Scope of Research

i. Parameters for carbonization screening process include weight of D(+)-Glucose (g), heating time (min) and power level of microwave (Watt).

ii. Sulfonation process parameters such as heating time (min), H_2SO_4 volume (ml), power level of microwave (Watt) and stirring rate (rpm) were fixed to obtain consistency throughout the screening process.

iii. Batch reactor connected to reflux condenser was used for esterification of PFAD, methanol and catalysts prepared by fixing the operating temperature (°C), reaction time (hr), molar ratio and catalyst loading (wt%) according to the optimum parameter of the previous study.

iv. The analysis of the methyl ester produced from esterification process was determined through GC-FID.

v. The selected catalysts from carbonization screening process were then characterized using FTIR, XRD, FESEM, BET and TPD-NH_3 to evaluate and analyze its chemical and physical properties.

vi. Optimization was conducted for sulfonation process of ICG using response surface methodology (RSM) to obtain the optimal operating conditions for sulfonation process using the best performing catalyst from carbonization screening process parameters.

vii. First order and second order kinetic study was conducted to seek for the reaction order, pre-exponential constant, and activation energy for the esterification process of PFAD to methyl ester using the optimized catalyst.

viii. The energy absorption by the catalyst prepared via microwave irradiation and conventional heating method as well as the cost analysis for catalyst synthesized via microwave irradiation and conventional heating method were determined.

1.6 Significance of Research

Biodiesel have attracted much attention as an alternative energy source since they are renewable, available locally, and have proved to be a cleaner fuel biodiesel. Palm oil is a common feedstock for biodiesel whereby a by-product called palm fatty acid distillate (PFAD) is usually generated during the refinery stage of the palm oil process. This study is significantly vital since it supports the renewable energy efforts where PFAD is fully utilized to be converted to methyl ester, an excellent option beside of using it for nutraceutical and cosmetic industry purpose. Traditionally, methyl ester are produced from palm oil, a well refined grade oil through transesterification process. However, the high cost of palm oil as a feedstock directly affects the high cost of methyl ester production. To make situation worsen, the fact that the biodiesel industry using palm oil is fighting against food supply is questioned. In fact, the preparation of catalyst for esterification process adopting microwave-assisted heating method is an alternative to save energy and time compared with conventional heating method. Common sulfonation or calcination of heterogeneous acid catalyst requires a lot of time with high temperature, thus by proposing a microwave-assisted method, the time of catalyst preparation can be shorten and low temperature can be applied and acquired for a large scale industry.

1.7 Organization of Thesis

The following chapters will be dedicated to carefully explain, discuss and analyze their respective topic in relation to the preparation of SO3H/ICG via microwave assisted heating method for esterification of PFAD.

Chapter 2 provides a comprehensive review on the topic of heterogeneous acid catalyst prepared via microwave-assisted heating method and conventional heating method. In the beginning, the chemistry of PFAD and its amazing economic potential as a renewable feedstock is elaborated. This is followed by the chemistry and importance of biodiesel or methyl ester as a potential sources for transportation. Since the esterification of PFAD required a very high acidity catalyst, an extensive

review of the technology of catalyst preparation via microwave and conventional method. The catalyst preparation include various type of catalyst based including the selected one which belongs to the carbon based group. Most importantly, the esterification of PFAD and methanol using the catalyst prepared via microwave-assisted heating method promotes green biodiesel, which is environmentally friendly as well as the potential of low cost of PFAD feedstock. This is followed by the discussion of the Design of Experiment (DOE) and Response Surface Methodology (RSM) tool that has been utilized for integrated process optimization of the process under study.

Chapter 3 explains the methodology of the experiment. A research methodology flow chart attached in the beginning of the chapter provides an overview of how the research is undertaken. The designing work is carried out cautiously, considering every aspect of catalyst development to experimental testing. It also includes justifications of why each materials or equipment is used in the esterification that has been used in the investigation. Catalyst characterization utilized in this study and its method is also included. Detail optimization methods on RSM, kinetic study and energy and cost analysis are incorporated as well.

Chapter 4 features the catalyst screening for the esterification of PFAD and methanol. One factor of a time (OFAT) was proposed to screen the parameters for carbonization process. 15 catalyst were prepared based on the OFAT and only five catalyst were selected namely A, B, C, D and E have been synthesized and carefully characterized using FESEM, BET, FTIR, TPD-NH_3 and XRD.

The catalyst were then catalytically tested for esterification of PFAD and methanol and the product were analyzed using GC-FID for yield and conversion percentage. TOF and regeneration of the catalysts was determined. D emerged as the best performing catalyst and the optimum parameter used for carbonization was used for further optimization process which is explained in further chapter.

Chapter 5 is dedicated to comprehensively deliberate on the optimization of the process using the best performing catalyst attained from previous chapter. Experiments were performed based on the arrangement proposed by RSM and two mathematical models correlating yield percentage (Y) and conversion percentage (C) with the operating parameter power level microwave, volume of H_2SO_4 (ml), heating time (min) and stirring rate (rpm) were produced. The accuracy of the models was statistically proven and the behavior and interaction among the independent variables were analyzed.

Chapter 6 explores the reaction kinetic of the catalyst prepared via microwave-irradiation to comprehend the fundamental details of the reaction. The order of reaction, activation energy and pre-exponential constant of the reaction was calculated. The reaction model denominator contains the product equilibrium constant being considered for the scenario under study. In this chapter also investigates the energy and cost analysis for the whole process from preparation of catalyst until esterification process of PFAD and methanol. The energy of catalyst prepared via microwave-assisted was then compared with catalyst prepared via conventional heating method. The cost analysis in the other hand was calculated based on laboratory scale, pilot plant and industrial plant for both microwave irradiation and conventional heating method.

Finally, Chapter 7 summarizes the primary conclusion of the present work, based on the results and findings obtained throughout the previous chapters. From the knowledge and experience attained in the present work, a list of useful recommendation is proposed for improvement in future work and its continuation opportunities.

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3. **Nur Nazlina Saimon,** Suwew Thavil, Mazura Jusoh, Norzita Ngadi, Zaki Yamani Zakaria. (2018). Preparation of Ti-SO3H/ICG using microwave-assisted for esterification of palm fatty acid distillate to methyl ester. Chemical Engineering Transaction. 63, 463-468. https://doi.org/10.3303/CET1863078**. (Indexed by SCOPUS)**

4. **Nur Nazlina Saimon,** Syafiq Isa, Yanti Maslina Jusoh, Mazura Jusoh, Norzita Ngadi, Zaki Yamani Zakaria. (2019). Optimization of esterification palm fatty acid distillate to methyl ester using microwave-assisted titanium sulfonated glucose acid catalyst. Chemical Engineering Transaction. 72, 367-372. https://doi.org/10.3303/CET1972062. **(Indexed by SCOPUS)**

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6. **Nur Nazlina Saimon,** Mazura Jusoh, Norzita Ngadi, Zaki Yamani Zakaria. (2020). Kinetic Study of Esterification of Palm Fatty Acid Distillate Using Sulfonated Glucose Prepared via Microwave-Assisted Heating Method. Chemical Engineering Transaction. 78, 403-408. https://doi.org/ 10.3303/CET2078068. **(Indexed by SCOPUS)**

7. **Nur Nazlina Saimon,** Norzita Ngadi, Mazura Jusoh, Zaki Yamani Zakaria. (2020). Optimization of Sulfonated Incomplete Carbonized Glucose Catalyst via Microwave-Assisted Heating for Biodiesel Synthesis from Palm Fatty Acid Distillate (PFAD). Bulletin of Chemical Reaction Engineering & Catalysis**. (Q3, IF:0.256)**