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CARBON-BASED CATALYSTS DERIVED FROM BIOMASS FOR THE PRODUCTION OF BIODIESEL FROM LOW QUALITY FATS

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By

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairman : Professor Datuk Taufiq Yap Yun Hin, PhD
Faculty : Science

Increase in awareness of the depletion of fossil duel resources makes biodiesel, a promising substitute of an alternative fuel. It is proved that biodiesel to be the best replacement for diesel because of its unique properties like significant reduction in greenhouse gas emissions, non-sulfur emissions, non-particulate matter pollutants, low toxicity and biodegradability. However, the cost of biodiesel is the major obstacle to its commercialization in comparison to petroleum-based diesel fuel. The high cost is primarily due to the raw material, mostly vegetable oil that are used as feedstock and utilization of catalyst. Thus in this study, biodiesel are studied using high free fatty acid feedstock (Chicken Fat and Skin Oil (CFSO) and Palm Fatty Acid Distillate (PFAD)). Simultaneous esterification and transesterification of chicken fat oil (CFSO) over Ce supported AC_{cs} catalyst that are treated using sulphuric acid and details study on the effect of Ce concentration from range 5 to 15 wt. % also further investigated. The results showed that 5wt. % Ce was an optimum concentration for optimizing the esterification and transesterification of CFSO with free fatty acid (FFA) conversion approximately 93%. For PFAD feedstock esterification reaction were conducted over chlorosulfonated treated carbon catalyst from various sources (Commercialized carbon (CC), Multi-Wall Carbon Nanotube (MWCNT) and carbon derived from coconut shell (CS)). Finding reveals FFA conversion increased followed the order of CCs>MWCNT>CSs. The biodiesel production both studies were optimized via one-variable –at-a –time (OVAT) by varying methanol to CFSO or PFAD, catalyst loading, reaction time and temperature. Optimization study using CFSO feedstock revealed at usage of 0.3wt% catalyst loading, 1:12 CFSO molar ratio to methanol within 1 hour reaction time at 90°C generated 93%. Whereas, chlorosulfonated carbon exhibited good catalytic activity with high FFA conversion of 95% at optimum parameter of 1:14 PFAD molar ratio to methanol, 0.5wt% of catalyst loading an 80°C reaction temperature within 2 hours reaction time.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KATALISIS BERASASKAN KARBON DARI BIOMAS UNTUK PENGELUARAN BIODIESEL DARI PENGGUNAAN LEMAK YANG BERKUALITI RENDAH

Oleh

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Peningkatan kesedaran mengenai kekurangan sumber fosil menjadikan biodiesel sebagai pengganti yang menjanjikan suatu bahan api alternatif yang mendapat perhatian luar biasa. Ia membuktikan bahawa biodiesel sebagai gantian terbaik untuk diesel kerana ciri-ciri yang unik seperti penggurangan yang ketara dalam pengeluaran rumah hijau gas, pelepasan gas bukan sulfur, bahan pencemar yang bukan zarah, ketoksikan rendah dan *biodegradability*. Walau bagaimanapun, kos biodiesel adalah halangan utama untuk mendagangkannya berbanding dengan bahan api diesel yang berasaskan petroleum. Kos yang tinggi adalah disebabkan oleh bahan mentah contohnya adalah minyak sayur-sayuran dan penggunaan pemangkin. Oleh itu dalam kajian ini, biodiesel dikaji menggunakan minyak yang lemaknya adalah tinggi (Minyak Lemak dan Kulit Ayam (CFSO) dan Palm Fatty Acid Distillate (PFAD)).Reaksi esterifikasi dan transesterifikasi serentak dari minyak lemak dan kulit ayam (CFSO) yang disokong oleh pemangkin Ce atas AC_{CS} yang dirawat menggunakan asid sulfurik dan kajian butiran mengenai kesan kepelbagaian dos Ce dari (5-15 wt. %) perlu dikaji. Manakala tindak balas pengesteran telah digunakan untuk PFAD sebagai bahan mentah dengan pemangkin karbon (karbon yang dikomersialkan (CC), Multi Wall Carbon Nanotube (MWCNT) dan karbon yang diperolehi dari tempurung kelapa (CS)) yang dirawat dengan asid khlorosulfonik. Kajian mendedahkan nilai FFA meningkat sejajar ; CCs> MWCNT> CSS. Pengeluaran biodiesel dari kedua-dua kajian telah dioptimumkan melalui satu pembolehubah -at-tidak- (ovat) dengan mengubah metanol CFSO atau PFAD, pemangkin loading, masa tindak balas dan suhu. kajian pengoptimuman menggunakan CFSO bahan mentah didedahkan pada penggunaan 0.3wt% pemangkin loading, 1:12 nisbah CFSO molar kepada metanol dalam masa tindak balas 1 jam pada 90 ° C yang

dihasilkan 93%. Manakala, karbon chlorosulfonated dipamerkan aktiviti pemangkin yang baik dengan tinggi FFA penukaran sebanyak 95% pada parameter optimum 01:14 PFAD nisbah molar kepada metanol, 0.5wt% daripada pemangkin memuatkan 80 ° C suhu tindak balas dalam tempoh 2 jam masa tindak balas.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Figure		Page

LIST OF ABBREVIATIONS

UPM	Universiti Putra Malaysia
FAME	Fatty Acid Methyl Ester
FT-IR	Fourier Transform Infrared
GC-FID	Gas Chromatograph Flame Ionization Detector
GC-MS	Gas Chromatograph Mass Spectrometry
TPD-NH_3	Temperature Programmed Desorption Ammonia
TPD-CO_2	Temperature Programmed Desorption Carbon Dioxide
FESEM	Field Emission Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction
XPS	X-Ray Photoelectron Spectroscopy
MWCNT	Multiwall Carbon Nanotube
CFSO	Chicken Fat and Skin Oil
CS	Coconut Shell
CC	Commercialized Carbon
ULSD	Ultra Low Sulfur Diesel

CHAPTER 1

INTRODUCTION

1.1 Energy Demand and Environmental Concern

A significant amount of primary and secondary energy sources is bequeathed to Earth for humans use. However, few of those primary energies which are fossil fuels and minerals are facing depletion time by time as they are largely consumed for vehicles as a fuel for human needs. Fossil fuels (coal, gas and oil) have and continue to play important role in the world energy market. According to (Oecd.org, 2020) world global primary energy demand is forecasted to increase on average by 1.46% per year from 2009 to 2035. (B. R. Singh & Singh, 2004) have reported 1.5 trillion dollars of world's energy market are powered by fossil fuels. Increase in the population growth resulting in total consumption of fossil fuels continues to increase which emanate in higher per capita energy demands, thus, simultaneously fluctuates the price of the fossil fuels. It is noteworthy to mention that continuous usage of fossil fuel also leads to the rise of greenhouse gases (GHG) such as CO_2 and simultaneously results in global warming issues (climate change) which in turn impacts the environment badly. It has been agreed that global warming causes tremendous pressure on environment such as glaciers melting in exponential rate and thus causing sea level to increase significantly. Figure 1.1 shows the climate change performance index for Malaysia. As observed from the graphs, the GHG emission are increasing year by year as the population and energy supply increases. In order to reduce the contributions to global warming and solve the issues of fossil fuel depletion, an alternative fossil fuel are urgently needed.

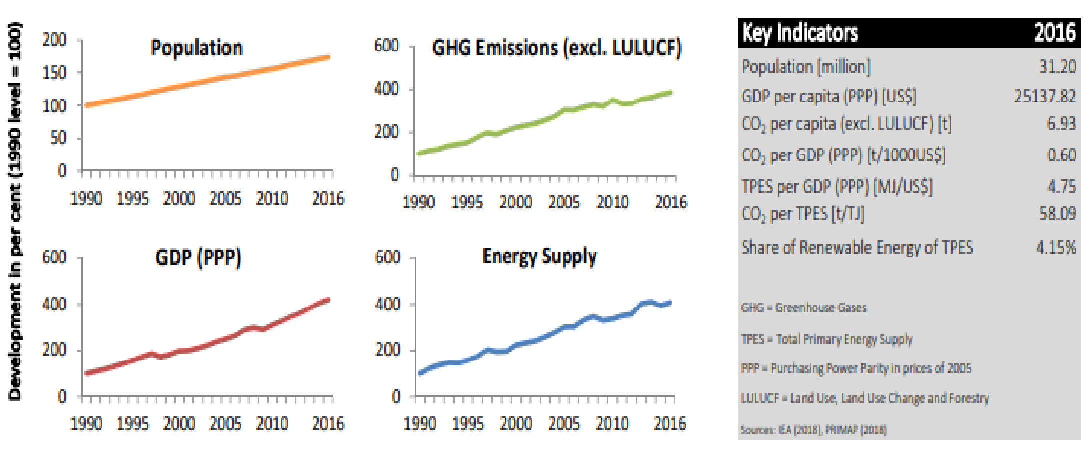

Figure 1.1 : Climate Change Perfomance Index in Malaysia
(Adapted from CCPI 2019)

1.2 Renewable Energy Derived from Biomass

Renewable fuels derived from biomass sources such as fats and oils, have received significant attention by many researchers as an alternative replacement for fossil fuel. Several production of renewable fuels using renewable feedstocks such as fats and oils have been studied enormously(Thushari *et al*., 2019) (Mardhiah *et al*., 2017). Biodiesel is the most well-established renewable fuels and biodiesel mainly consists of fatty acid methyl esters (FAME) (Sajjadi *et al*., 2016). Chemically, biodiesel is defined as the mono alkyl esters of long chain fatty acids, and typically produced through the reaction such as transesterification and esterification of a vegetable oils or animal fats with methanol or ethanol in the presence of catalyst. The usage of biodiesel as an alternative fuel for diesel engine have been reported (Mardhiah *et al*., 2017) and it was discovered that the use of biodiesel in the diesel engine is beneficial for environment. Biodiesel combustion typically emitted less COx and NOx emission than petroleum diesel combustion (Atadashi *et al*., 2013) . This is due to its composition of biodiesel in which typically made up of 10-11% oxygen, nitrogen and sulfur-free (Ayhan, 2007; Silitonga *et al*., 2013). It have been reported by Mofijur and co-worker that the oxygenated species in biodiesel could improve the combustion in engines and reduce the fuel's oxidation potential (Mofijur *et al*., 2014). Currently, biodiesel gives positive impact as the alternative resources for a better future in mankind. Evidently, according to Global Agricultural Information Technology, in the year 2019, biodiesel production is expected to increase by approximately 36% compared to previous years to 1.69 billion liters due to the increase in the domestic use and exports (Lumpur *et al*., 2019). Recently, Malaysia as world's second largest producer of palm oil have explored the production of biodiesel from palm oil and have successfully commercialized the biodiesel in the blending formation known as B5 (95% of petroleum diesel: 5% of biodiesel) . These B5 has been sold each month at 247 petrol stations in Malaysia as can be seen in Figure 1.2.

Figure 1.2 : Regions of B5 Sale in Malaysia
(Adapted from Asia Biomass Office)

1.3 Chicken Fat Skin Oil CFSO) and Palm Fatty Acid Distillate (PFAD)

Biodiesel can be acquired from a variety of sources including vegetable oils, animal fats, and waste cooking oil (WCO). Due to large availability of vegetable oil, typically the vegetable oil such as palm oil, corn oil, sunflower oil, soybean oil and rapeseed oil are highly preferred oil for production of biodiesel (Lam *et al*., 2009). However, the usage of edible vegetable oil as biodiesel feedstocks create a food versus fuel conflict and thus it usage in biodiesel production shall result in disturbing of food security. Moreover, the high prices of commodity edible vegetable oils become another drawback to the usage of edible oil as biodiesel feedstock. Due to this reason, it is essential to find potential economical feedstocks and do not compete with food chain. Inedible oils such as palm fatty acid distillate (PFAD), animal fats (such as chicken fat, beef tallow and etc.) WCO and by-products of the refining vegetable oil are few examples of less expensive and easily available non-edible feedstocks. Among those, nowadays chicken fat and skin (CFS) and (PFAD) are gaining attention by many researches.

Majority of CFS and PFAD comprised of high oil content and majority of the these feeds contain high free fatty acid (FFA), whereas, CFS consist of 5–30% FFA, while PFAD consist of 85% FFA, respectively (Lokman *et al*, 2016). CFS is highly consumed by humans since it may give better flavor in many cuisine, Yet, this CFS have more oil content and is comprised of more unsaturated fatty acid (55-60%), thus it is not good for human health. Since these animal fats are not suitable for human food chain and create huge amount of waste from the large food processing,it can therefore be a great opportunity to obtain biodiesel from CFS. PFAD is a by-product of low market value which itself is a product from refined palm oil. It consists of a mixture of FFA and other minor components like mono, di and triglycerides and unsaponifiable matters (Saravanan *et al*., 2019). Taking advantage on the availability of PFAD as feedstock would provide economic advantage rather than using high-value edible palm oil. PFAD is currently used in industry as boilers, raw material for oleochemical industries and as animal feed ingredient.

Since CFS and PFAD is high FFA oil (>5%), acid catalyst required to convert those FFA to biodiesel. Many studies have used zeolites-based (Mohammadi *et al*., 2015), alumina-based (Adeyinka Sikiru Yusuff & Owolabi, 2019) and zirconia-based catalyst (Luo *et al*., 2017) in biodiesel reaction. However due to high cost and catalyst stability problem (Alcañiz-monge *et al*., 2018) , researchers urge to find a promising catalyst that can help to solve this issue. Carbon-based catalyst have received attention due to its advantages of low cost, abundance, chemical inertness, mechanical stability and high catalytic performance (Xincheng *et al*., 2019). Eventhough carbon-based catalyst is very promising in biodiesel production reaction, yet it still required more acidic sites for motivating the FFA conversion to biodiesel. Sulfonation and chlorosulfonation treatment on carbon have been established and widely accepted as the best techniques for improving the carbon-based catalyst properties. Both acid treatment capable in functionalizing the carbon materials with strong acid groups (Carvalho *et al*., 2020) and it was revealed that both acid treatment resulted in higher acidity. Recently reported that these sulfonated and chlorosulfonated carbon catalysts were found to be effective in converting FFA toward biodiesel (Yadav & Murkute, 2004).

1.4 Problem Statement

Rapid growths in population, industrialization and urbanization have resulted in continuous need of energy. Even though fossil fuels are the world's primary source of energy, this source has limits and begins to worry the society. In addition to it, continuous consumption of fossil fuel had increased CO_2 emissions, which is responsible for the greenhouse effect. Thus a significant demand for renewable energy derived from renewable sources and at the same time is not harmful to the environment is becoming a high priority (Fontoura *et al*., 2019). Biodiesel production industry has gradually grown as an alternative to replace fossil fuel consumption. Unfortunately, high cost production especially for the biodiesel feedstock and process of product purification have become major barrier. Thus, in the current study we will highlight the usage of cheaper and waste feedstock derived chicken fat and skin oil (CFSO) and palm fatty acid distillate (PFAD) and develop an effective inexpensive heterogeneous acidic catalyst for converting high FFA feedstocks; CFSO and PFAD along for lowering the biodiesel production cost. In order to achieve a high conversion in biodiesel, a good catalyst is required.

Homogenous catalyst is sensitive to water and FFAs which can cause soap formation if too much catalyst is used. This decreases the biodiesel yield and causes problem during product purification especially while generating huge amount of wastewater. Use of carbon-based catalysts in these reactions opens doors for cost minimization and environmentally benign biodiesel production by eliminating problems associated with the conventionally used reaction schemes (employing homogeneous catalysts commonly H2SO4, KOH or NaOH). As reported by different researches owing to its high thermal stability, unique surface and structural properties it can be utilized as a support for the variety of active catalysts (metal, metal oxide and so on) with the possibility to be utilized as heterogeneous catalysts in different reactions including biodiesel synthesis. But, the acidic carbon catalyst still suffered with catalyst instability (low reusability), thus in order to improve the strength of the bonding of acidic functionalized species on carbon, herein the present study, various carbons from different sources will be activated using concentrated sulfuric acid and chlorosulfonic acid. Since CFSO also showed the presence of triglycerides, we are also developing acid-base carbon catalyst for motivating simultaneous esterification and transesterification process.

1.5 Objectives

The objectives of this research are listed as below:

1. To synthesize and characterize heterogeneous sulfonated and chlorosulfonated carbon catalysts.
2. To investigate the esterification and transesterification of low quality fats (CFSO and PFAD) over heterogeneous sulfonated and chlorosulfonated carbon catalysts respectively.
3. To optimize the esterification and transesterification reaction conditions for biodiesel reaction via one-variable-at-a- time (OVAT) method.
4. To study the stability and reusability of the catalyst.
5. To evaluate and determine the fuel properties of CFSO biodiesel.

1.6 Scope of research

In this study, the catalyst is prepared from coconut shell and cerium (III) metal precursor via sulfonation processes. The catalyst produced undergoes characterization by XRD, TPD-NH3, TPD –CO2, FTIR, TGA, BET and XPS. The catalytic performance for all catalysts were screen and determined at certain condition parameter in order to choose the best catalyst with high catalytic performance for the optimization study. The effect of the molar ratio of methanol to oil, catalyst loading, reaction temperature and reaction time were investigated by conducting simultaneous esterification and transesterification reaction of CFSO. The methanol ratio that been used 12:1 to 24:1. Meanwhile, the temperature that use is 50 – 100°C and time from 30 minutes -2h 30 minutes was varied with catalyst loading form 1-5wt%. Reusability test is a test applied on catalyst where several cycles of reaction conducted by using the same catalyst without modifying the catalyst and the amount of sulfur content was determined by using XPS analysis. The CFSO biodiesel properties was determined by using ASTM D6751 and EN 14212 standard. The optimized catalyst was also further used in esterification of PFAD. It is known that esterification reaction requires acidic catalyst thus carbon catalyst that are treated with (chlorosulfonic acid) were used without cerium metal. The catalytic performance for all catalysts were screened and determined at certain condition parameter in order to choose the best catalyst with high catalytic performance for the optimization study. The effect of the molar ratio of methanol to oil, catalyst loading, reaction temperature and reaction time were investigated by conducting simultaneous esterification and transesterification reaction of CFSO. The methanol ratio that been used 6:1 to 22:1. Meanwhile, the temperature that use is 60 – 100°C and time from 30 minutes - 6 hour was varied with catalyst loading form 0.1-5wt%.The biodiesel produced was analysed using GCMS.

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