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CATALYTIC PYROLYSIS OF CHICKEN FAT WASTE USING ZEOLITE CATALYSTS FOR BIODIESEL PRODUCTION

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science.

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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.

CATALYTIC PYROLYSIS OF CHICKEN FAT WASTE USING ZEOLITE CATALYSTS FOR BIODIESEL PRODUCTION

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July 2014

Chairman: Assoc. Prof. Salmiaton binti Ali, PhD

Faculty: Engineering

Chicken fats become a major poultry waste from broiler industries and the production rate is increasing with human population. The conversion of waste chicken fats containing major triglycerides into potential biodiesel was investigated under non-catalytic and catalytic pyrolysis process using ZSM-5, HY and FCC zeolite catalyts (0.2-1.0 wt. %). Pyrolysis was conducted using lab-scale distillation set-up upon the variation of temperature, reaction time and nitrogen flow rate from 325-425°C, 10-50 min and 100-500 ml/min, respectively, to study the effect on the production of tar, pyro-oil and gas. Based on the analysis, increase of temperature, reaction time and nitrogen flow rate caused an increase of pyro-oil and gas, and a decrease of tar. This phenomena happened as a result of efficient thermal decomposition of biomass with excessive retention time led to an increase to the production of pyrolyzed gas and formation of pyro-oil. The maximum yield of pyro-oil (73.81%) occurred at temperature 425°C, 30 minutes under nitrogen flow rate of 300 ml/min producing tar and gas at 17.57% and 8.62%, respectively. From catalytic pyrolysis, 0.8% loading of ZSM-5 and FCC, and 0.6% loading of HY produced the maximum yield of pyro-oil at which FCC showed the greatest increment at 18.97%. Pyro-oils were further analysed by Gas Chromatography-Mass Spectroscopy (GCMS) and Fourier-Transform Infrared Spectroscopy (FTIR) in order to identify their chemical properties and functional groups. From the GCMS analysis, different short hydrocarbon chain products in the range of C_4 – C_{31} were found with dominant by chemical class of aliphatic, followed by carboxylic acid, cycloaliphatic, aromatic, ketone, ester and anhydride. The GCMS results were supported by related functional groups determined by FTIR, which are O-H hydroxyl group stretch, C=O carbonyl group stretch, C-O stretch, C-H aliphatic stretch, -CH_2- aliphatic stretch and C-H bending (out-of-plane). Zeolites showed their selectivity in the production of aliphatic, cycloaliphatic and aromatic. ZSM-5 displayed the greatest increment of aliphatic (16.06%) and aromatic (1.79%). Meanwhile, FCC showed the greatest influence in reducing carboxylic acid by 16.15%. The presence of catalysts also affected the physical properties with the decrease of density, viscosity, acid value,

saponification value, flash point, cloud point and pour point, and the increase of calorific value. As compared to Brazilian diesel fuel specification and ASTM D6751 standard, pyro-oils adequately met the qualifications except their acid values were out of the range of limit. Additionally, catalytic pyro-oils found high in calorific value had showed its commercial value to use in supplying heat and electricity for vehicle engine, burner, broiler and stirring engine.

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

PEMANGKINAN PIROLISIS SISA LEMAK AYAM DENGAN MENGGUNAKAN PEMANGKIN ZEOLIT UNTUK PENGHASILAN BIODIESEL

Oleh

LIEW MEI YI

Julai 2014

Pengerusi: Professor Madya Salmiaton binti Ali, PhD

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Lemak ayam merupakan sisa ternakan utama dari industri daging ayam dan kadar pengeluarannya semakin meningkat dengan populasi manusia. Penukaran sisa lemak ayam yang mengandungi terutamanya trigliserida ke biodiesel yang berpotensi telah disiasat menggunakan proses pirolisis dengan tiada pemangkin dan dengan pemangkin zeolit ZSM-5, HY dan FCC (0.2-1.0 wt. %). Pirolisis telah dijalankan dengan menggunakan set-up penyulingan berskala makmal atas perubahan suhu, masa tindak balas dan kadar aliran nitrogen dari 325-425°C, 10-50 min dan 100-500 ml/min masing-masing, untuk mengkaji kesan pengeluaran tar, piro-minyak dan gas. Menurut analisis, peningkatan suhu, masa tindak balas dan kadar aliran nitrogen menyebabkan peningkatan pada piro-minyak dan gas, dan penurunan pada tar. Fenomena ini diakibatkan oleh penguraian terma pada biomas yang cekap dengan masa tindak balas yang berlebihan membawa kesan peningkatan pada pengeluaran pirolisis gas dan pembentukan piro-minyak. Penghasilan maximum piro-minyak (73.81%) berlaku pada suhu 425°C dan masa 30 min di bawah kadar aliran nitrogen 300 ml/min dengan pengeluaran tar dan gas pada 17.57% dan 8.62% masing-masing. Pada pirolisis pemangkinan, muatan ZSM-5 dan FCC pada 0.8%, dan muatan HY pada 0.6% menghasilkan maksimum piro-minyak, di mana FCC menunjukkan peningkatan tertinggi pada 18.97%. Piro-minyak telah dianalisis oleh Kromatografi Gas Spektroskopi Jisim (GCMS) dan Fourier-Ubahan Spektroskopi Inframerah (FTIR) untuk mengenalpasti sifat-sifat kimia dan kumpulan berfungsi. Daripada analisis GCMS, produk hidrokarbon berantaian pendek yang berbeza-beza didapati dari C_4-C_{31} dengan kelas kimia didominan oleh alifatik, diikuti oleh asid karboksilik, sikloalifatik, aromatik, keton, ester dan anhidrida. Keputusan GCMS disokong oleh kumpulan berfungsi berkaitan yang ditentu dalam FTIR, iaitu kumpulan regangan O-H hidroksil, regangan C=O kumpulan karbonil, regangan C-O, regangan C-H alifatik, regangan -CH_2- alifatik dan C-H bengkok (luar dari satah). Zeolit menunjukkan selektiviti mereka dalam pengeluaran produk alifatik, sikloalifatik dan aromatik. ZSM-5 memaparkan kenaikan tertinggi pada kumpulan alifatik (16.06%) dan aromatik (1.79%). Manakala, FCC menunjukkan pengaruh yang terbesar dalam

mengurangkan asid karboksilik pada 16.15%. Kehadiran pemangkin juga memberi kesan kepada sifat-sifat fizikal dengan penurunan pada ketumpatan, kelikatan, nilai asid, nilai penyabunan, takat kilat, titik awan dan titik curah, dan peningkatan pada nilai kalori. Berbanding dengan spesifikasi diesel Brazil dan piawai ASTM D6751, piro-minyak secukup-cukupnya memenuhi kelayakan kecuali nilai asid luar dari julat. Selain itu, pemangkinan piro-minyak didapati tinggi pada nilai kalori telah menunjukkan nilai komersialnya dalam membekalkan haba dan elektrik untuk enjin kenderaan, pembakar, dandang dan enjin kacau.

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I certify that a Thesis Examination Committee has met on 22nd July 2014 to conduct the final examination of Liew Mei Yi on her thesis entitled "Pyrolysis of Waste Chicken Fats Using Zeolite Catalysts for Biodiesel Production" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the degree of Master of Science.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
AV	Acid Value
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halena (Bjh)
DCM	Dichloromethane
DV	Dynamic Viscosity
FAME	Fatty Acid Methyl Ester
FCC	Fluid Catalytic Cracking
FTIR	Fourier-Transform Infrared
GCMS	Gas Chromatography-Mass Spectroscopy
HDPE	High-Density Polypropylene
HHV	Higher Heating Value
HZSM-5	Proton Exchange Zeolite Socony Mobil-5
KV	Kinematic Viscosity
LDPE	Low-Density Polypropylene
LHV	Lower Heating Value
PE	Polyethylene
PET	Polyethylene Terephthalate
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl Chloride
SEM-EDX	Scanning Electron Microscope-Energy Dispersive X-Ray
SV	Saponification Value
TGA-DTG	Thermogravimetric Analysis-Differential Thermogravimetry
XRD	X-Ray Diffraction
ZSM-5	Zeolite Socony Mobil-5

CHAPTER 1

INTRODUCTION

1.1 Energy Resources

Energy resources can be referred to fossil, renewable and fissile energy. Petroleum is known as current worldwide largest energy resource as it provides massive energy sources, surpassing coal, natural gas, nuclear, hydro, and renewable (Sarin, 2012). Based on the estimation of global oil production (Figure 1.1), the international scarcity of these conventional fossil fuels is forecasted after the peak production (2015-2030) and this issue will lead to an oil crisis due to the depletion of reserves (Ayhan, 2009). Therefore, renewable energy becomes a compromising alternative solution to resolve the international scarcity of fossil fuel issues and its high emission of greenhouse gasses (Elvers, 2008).

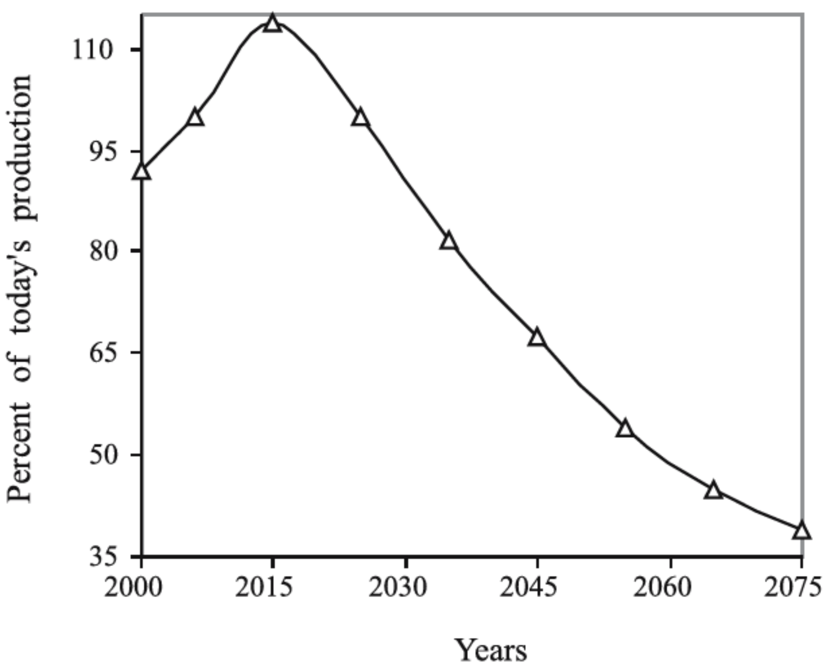

Figure 1.1 Scenarios of global oil production based on current production (Ayhan, 2009)

Biomass energy is kind of renewable energy that involves thermochemical, biological or chemical conversion of biomass into different kinds of energy sources such as solid, gaseous and liquid fuels (Maher and Bressler, 2007). Current alternative liquid fuels for transportation are ethanol and biodiesel. The main feedstock of ethanol are sugar or starch crops that accounts for total biofuel production at approximate 86%, and the rest of it accounts to biodiesel that produced from vegetable oils or animal fats. The availability of biomass for the biofuel productions is investigated especially waste crops and low-cost residues are highly preferred for sustainable bio-energy production (Khanal et al., 2010).

By general definition, biodiesel is fatty acid methyl ester produced from transesterification. Biodiesel also refers to a diesel-equivalent or diesel-like property biofuel that produced from organic oils and fats (Demirbas, 2009a). The diesel-like bio-oil can be produced using different biomass conversion techniques such as pyrolysis, gasification and thermal cracking (Laksmono et al., 2013; Lin et al.; 2013, Tang et al.; 2012).

The property of biodiesel is highly competitive to fossil diesel due to more environmental benefits are provided such as non-toxicity, biodegradable, suitable for sensitive environments, renewable and energy efficient, replaceable for petroleum-derived diesel fuel, low greenhouse gasses emission, and it is made from agricultural or recycled resources (Wang et al., 2011). However, biodiesel shows weaker operating properties in vehicle engine as compared to petrol diesel due to its higher viscosity, lower volatility and higher concentration of unsaturated hydrocarbons (Savaliya et al., 2013).

There are two types of biodiesel applied in diesel vehicle engine namely straight biodiesel (B100) and blending biodiesel (B5 to B20). The lower portion of biodiesel blends releases lower amounts of carbon oxides and nitrogen oxides gases. This makes B20 more commonly used in the US and European countries (EU) in order to cut off the consumption of diesel fuel (Worldwatch Institute, 2012; Demirbas, 2009a). And, the growing of biodiesel consumption associates with the steady increase of biodiesel production (Figure 1.2), especially in EU where further leads to a growing in the development of biodiesel compatible equipment and engine (Licht, 2012).

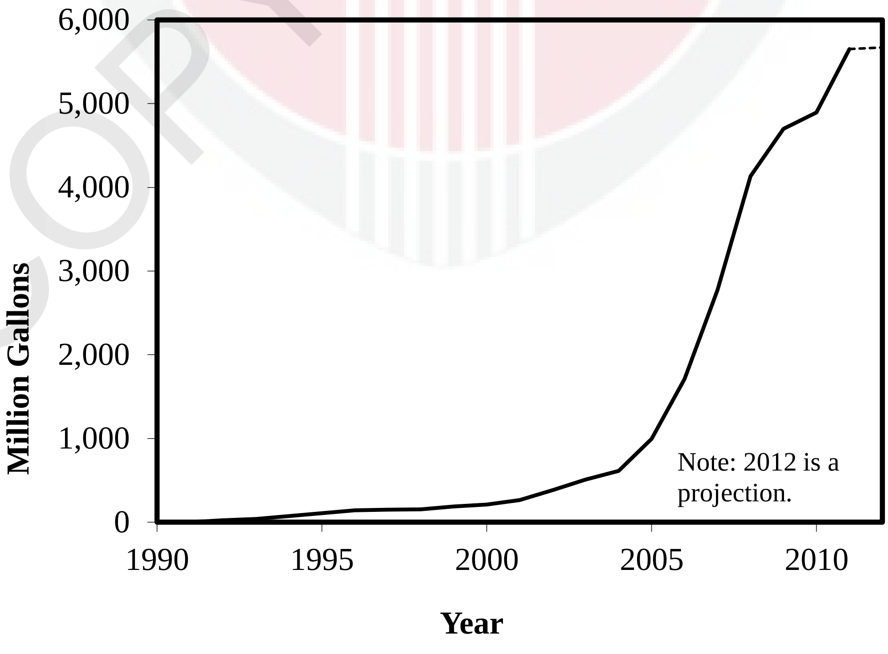

Figure 1.2 World biodiesel productions from 1991 to 2012 (Licht, 2012)

1.2 Biomass Feedstock for Biodiesel Production

The nature, type and composition of biomass can directly affect the biodiesel properties. Generally, biodiesel produced from vegetable oils and animal fats comprises mainly triglycerides that plays important role in chemically conversion (Canakci and Van Gerpen, 2001). The qualities of biodiesel are directly manipulated by the degree of fatty acids caused it influences the performance characteristics such as flow properties and volatility (Sengupta and Pike, 2012).

1.2.1 Vegetable Oils

Vegetable oils provide primarily first generation feedstock in biodiesel production. Table 1.1 shows the major fatty acids distribution of various vegetable oils. Soybean oil is the major interest of biodiesel source in the US. European, on the other hand, prefers rapeseed oil at which produces approximate 70% of biodiesel from rapeseed. In Asia, for instance, palm oil is the dominant crops in Malaysia that potentially used to made biodiesel due to its high content of triglycerides and low acid value (Khanal et al., 2010; Elvers, 2008).

The good points of using vegetable oils as biodiesel are easy accessibility of crops, low sulfur content and low sulfur oxide emission, fuel properties similar to petroleum diesel, and renewable. But, the major disadvantages of vegetable derived biodiesel are their higher density and viscosity, and lower volatility compared to petroleum diesel (Nag, 2007). Those bad properties affect the engine operation causing coke deposition and trumpet formation on the injector and gelling in the engine (Demirbaş, 2003). From Table 1.2, sunflower oil, cottonseed oil and soybean oil derived biodiesel give better combustion character than corn oil, opium poppy oil and rapeseed oil due to their higher heating value and cetane number (Ayhan, 2009).

Table 1.1 Fatty acids distribution of vegetable oils (Demirbas, 2008)

Source	16:0	16:1	18:0	18:1	18:2	18:3	Others
Palm	42.6	0.3	4.4	40.5	10.1	0.2	1.1
Cottonseed	28.7	0.0	0.9	13.0	57.4	0.0	0.0
Rapeseed	3.5	0.0	0.9	64.1	22.3	8.2	0.0
Soybean	13.9	0.3	2.1	23.2	56.2	4.3	0.0
Coconut	9.7	0.1	3.0	6.9	2.2	0.0	65.7
Almond kernel	6.5	0.5	1.4	70.7	20.0	0.0	0.9
Sunflower seed	6.4	0.1	2.9	17.7	72.9	0.0	0.0

Table 1.2 Comparison of vegetable derived biodiesel (Demirbas, 2008)

Property	Sunflower	Cotton seed	Soybean	Corn	Opium poppy	Rapeseed
Heating value (MJ/kg)	39.5	39.6	39.6	37.8	38.9	37.6
Cetane No.	37.1	48.1	38.0	37.6	-	37.6

1.2.2 Animal Fats

Animal fats contain high fatty acids (Table 1.3), but more saturate than vegetable oils. Higher saturation causes higher cetane number, weaker cool flow property and lower lubricity. It explains that animal fat based biodiesel exhibits higher cetane number and combustion character due to higher ratio of carbon to hydrogen (C/H). Besides, it gives a better oxidative stability aspect than vegetable oil due to higher amount of saturated fatty acid presence (Knothe, 2005). However, high saturation of fatty acid in animal fats caused the animal fat derived biodiesel is more viscous than soybean based biodiesel as presented in Waytt et al. (2005) research.

Table 1.3 Fatty acids distribution of animal fats (Wyatt et al., 2005)

Source	14:0	14:1	15:0	16:0	16:1	17:0	18:0	18:1	18:2	18:3
Tallow	3.1	1.3	0.5	23.8	4.7	1.1	12.7	47.2	2.6	0.8
Lard	1.3	0.0	0.0	23.5	2.6	0.4	13.5	41.7	10.7	0.0
Chicken fat	0.7	0.13	0.0	20.9	5.4	0.0	5.6	40.9	20.5	0.0

The importance of animal fats as biodiesel feedstock arises due to the high consumption of vegetable oils in the first generation biofuel production that being conflict with the food supply. The competition of crops such as grain, plant seed, corn and sugar with biofuel production increase the price of crops and foods (Savaliya et al., 2013). Non-edible oil, like animal fats, is another preferred source in the biodiesel production that competitive to vegetable oil due to abundant of waste animal fats which can be immediate accessed with low cost price in slaughterhouse or livestock producing country, such as Southern Brazil (Cunha Jr et al., 2013).

1.3 Problem Statement

The importance of renewable energy, such as biodiesel has arisen due to the increase of the price of fuel diesel and the depletion of the coal reserves. The biodiesel production using pyrolysis process considers as a potential process for producing diesel-like-property biodiesel due to it contains high calorific value and cetane number as similar to diesel fuel properties (Sarin, 2012).

However, current research of pyrolysis reaction was more focus on vegetable oils (e.g. palm oil, sunflower seed, grape seeds and soybean) and cellulosic basis plants due to the ease of accessibility. There was lack of study on animal fats conversion using pyrolysis although the animal fats contains high triglycerides that can be converted into biodiesel as the same as vegetable oils. The research of pyrolysis on animal fats was limited on the feedstock of lard, tallow and fish oil. While, chicken fats was found not yet been studied or published by anyone as a feedstock of pyrolysis.

In Malaysia, chicken meat is the largest consumption among the poultries as in worldwide as well (FAO, 2011). The growing of chicken meat industries generate abundant of fat wastage that can be used as a potential source of hydrocarbon or biodiesel by chemically conversion such as pyrolysis. The waste can be transformed into profitable products, and at the same time reduce the cost of waste management.

Therefore, chicken fats become the target source of this research in order to discover their potential biodiesel value. Besides, this research also helped to extend the pyrolysis research field using animal fats feedstock with providing a new potential source for biodiesel production.

1.4 Objectives

The main purpose of this research is to investigate the pyrolysis of waste chicken fats in non-catalytic and catalytic conditions using zeolite catalysts, and determine the yield production, chemical compositions and physical properties. Besides, the effectiveness of fresh and used zeolite catalysts toward yield is also one of the concerns to verify the reactivity of the zeolite catalysts in recycle or reuse production. The specific objectives of this research are set as shown below:

a. To determine optimum conditions for pyrolysis of waste chicken fats into pyro-oil production.
b. To evaluate the effectiveness of ZSM-5, HY and FCC zeolite catalysts in pyrolysis using waste chicken fats before and after recovery.
c. To characterize chemical and physical properties of non-catalytic and catalytic pyro-oils.

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