OPTIMIZATION AND KINETIC MODEL FOR TRANSESTERIFICATION OF WASTE COOKING OIL USING TUNGSTOPHOSPHORIC ACID CATALYST

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TO MY BELOVED WIFE,

MY LOVING MOTHER AND MY BROTHER

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ABSTRACT

Transesterification of waste cooking oil with heterogeneous tungstophosphoric acid (TPA) catalyst and methanol was investigated. Response Surface Methodology (RSM) and Artificial Neural Network (ANN) were employed to study the relationships of process variables on free fatty acid conversion and for predicting the optimal conditions. The highest conversion was 88.6% at optimum reaction conditions of 65 ˚C reaction temperature, 70:1 molar ratio of methanol to oil, 10 %wt catalyst amount, and 14 h reaction time. The RSM and ANN could accurately predict the experimental results, with R^2 = 0.9987 and R^2= 0.985, respectively. The TPA catalyst exhibited good potential as a stable and active catalyst over four time’s reusability. The reaction followed first-order kinetics and the calculated activation energy was Ea= 53.99 kJ/mol while the pre-exponential factor was A= $2.9x10^7$ min^{-1}. According to the biodiesel characterization results (ASTM D6751) the product of this study has high potential for using as a substitute for conventional catalyst in biodiesel production.

ABSTRAK

Transesterifikasi minyak masak terpakai dengan mangkin heterogen tungstophosphoric acid (TPA) dan metanol telah dikaji. Metodologi permukaan bertindakbalas (RSM) dan rangkaian neural tiruan (ANN) digunakan untuk mengkaji hubungan antara pemboleh ubah proses dan penukaran asid lemak bebas dan untuk mengkaji parameter optimum. Penukaran yang paling tinggi ialah dengan kadar 88.6 peratus, dengan keadaan optimum 14 jam masa tindak balas, suhu tindak balas 65 darjah Celcius, nisbah molar methanol dan minyak 70:1 dan 10% kepekatan mangkin. RSM dan ANN boleh meramal keputusan eksperimen dengan tepat; R^2 bagi RSM ialah 0.9987 dan R^2 bagi ANN ialah 0.985. Mangkin TPA mempunyai potensi tertinggi sebagai mangkin yang aktif dan stabil dan boleh digunakan berulang kali. Tindak balas ini mematuhi kinetik tertib pertama dan tenaga pengaktifan ialah Ea = 53.99 kJ/mol; factor pra-eksponen sama dengan A= $2.9x10^7$ min^{-1}. Kajian ini dapat menghasilkan proses penghasilan biodisel dengan minyak masak terbuang dengan mangkin TPA heterogen yang mesra alam dan menepati piawaian ASTM D6751.

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LIST OF ABBREVIATIONS

ABE	Antivated Bleaching Earth
ANN	Artificial Neural Network
COME	Canola Oil Methyl Ester
DG	Diglycerides
EBP	End Boiling Point
E	Ester
FAME	Fatty Acid Methyl Ester
FFA	Free Fatty Acid
GL	Glycerol
KF	Potassium Fluoride
KOME	Karanja Oil Methyl Ester
MG	Mono Glycerides
PAH	Polycyclic Aromatic Hydro
PKOME	Palm Kernel Oil Methyl Ester
PPOME	Palm Oil Methyl Ester
PME	Palm Oil Methyl Ester
RSM	Response Surface Methodology
ROME	Rice Bran Oil Methyl Ester
SOME	Soybean Oil Methyl Ester
SFOME	Sunflower Oil Methyl Ester
TG	Triglyceride
THF	Tetra Hydro Furan

LIST OF SYMBOLS

R^2_{adj}	Adjust R-Square
Ea	Activation Energy (kJ/mol)
β_0	Constant Coefficient
X	Conversion (%)
ε	Error
R	Gas Constant (m^3. Pa/ K. mol)
C_{A0}	Initial Concentration
A	Pre-Exponential Factor
y_{pi}	Predicted Out Put Values
η	Response
R^2	R-Square
k	Reaction Rate Constant (1/min)
ra	Reaction Rate
t	Time (h)

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Energy consumption is inevitable for human existence. There are various reasons for the search of an alternative fuel that is technically feasible, environmentally acceptable, economically competitive, and readily available. The first and most important reason is the increasing demand for fossil fuels in all sectors of human life, be it transportation, power generation, industrial processes, and residential consumption. This increased demand gives rise to environmental concerns such as increased CO_2 emission, production of greenhouse gases, and global warming. World energy consumption doubled between 1971 and 2001 (Figure 1.1) and the world energy demand will increase 53% by the year 2030. For instance, petroleum consumption will rise from 84.4 to 116 million barrels per day in USA until year 2030 (Bioengineering Resource; Energy Department).

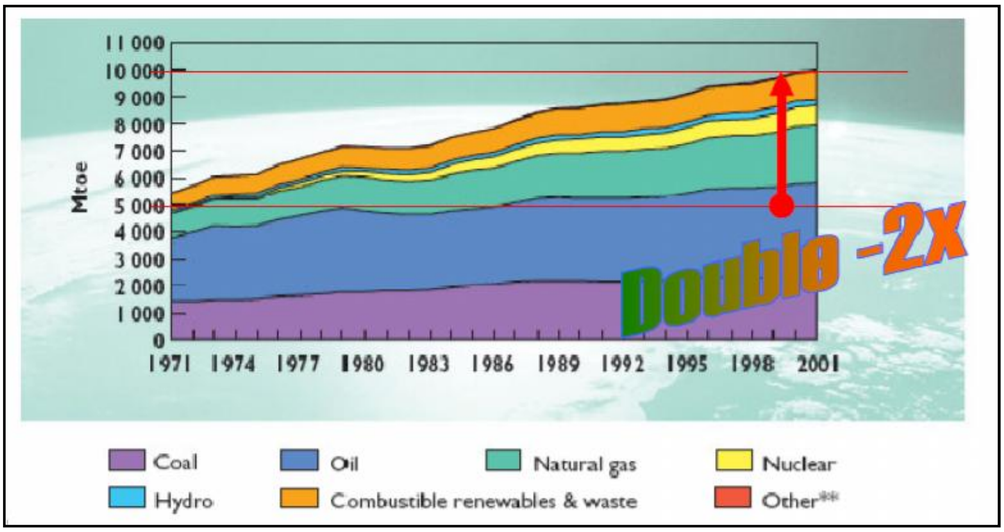

Figure 1.1 World energy supplies

The second reason is that fossil fuel resources are non renewable, and they will be exhausted in the near future. Some reports show that oil and gas reserves will be depleted in 41 and 63 years, respectively, if the consumption pace remain constant (Reports and publications). The last reason is the price instability of fuels such as crude oil, which is a serious threat for countries with limited resources. Several alternatives such as wind, solar, hydro, nuclear, bio-fuel, and biodiesel have been suggested but all of them are still in the research and development stage.

The inventor of biodiesel engines, Rudolf Christian Karl Diesel (1858-1913) demonstrated the use of vegetable oils as a substitute for diesel fuel in the 19th century (Orchad *et al*., 2007). He believed the utilization of biomass fuel will becomes a reality as future versions of his engine are designed and developed.

Biodiesel is a mono alkyl ester of fatty acids produced from vegetable oils or animal fats. In other words, when a vegetable oil or animal fat chemically reacts with an alcohol, it can produce Fatty Acid Methyl Ester (FAME); a vegetable oil can be used in diesel engines after some adjustments and modifications. Vegetable oils contain saturated hydrocarbons (triglycerides) which consist of glycerol and esters of fatty acids. In addition, fatty acids have different numbers of bonds and carbon chain lengths. There are different kinds of modification methods, such as dilution, thermal cracking (pyrolysis), transesterification, and microemulsification. However, transesterification is the best method for producing higher quality biodiesel (Meher *et al*., 2006; Knothe, 2001; Sinha *et al*., 2008; Ramadhas *et al*., 2004; Sharma and Singh, 2008).

All fatty acid sources such as animal fats or plant lipids (more than 300 types of them) can be used in biodiesel production (Basha *et al*., 2009; Canoira *et al*., 2006; Keshin *et al*., 2008). Table 1.1 show various feedstock's in biodiesel production (Ejas and Younis, 2011). The utilization of these types of sources has given rise to certain concerns as some of them are important food chain materials (Pimentel *et al*., 2009; Srinivasan, 2009). In other words, the production of bio-fuels from human nutrition sources can cause a food crisis. Therefore, the majority of researchers have used non-edible oils or waste edible oils as feedstock for biodiesel production. The most important obstacle in biodiesel industrialization and commercialization is production costs. Therefore, the usage of waste edible oils can reduce biodiesel production costs by 60% to 90% (Canakci and Van Gerpen, 2001; Zhang *et al*., 2003b; Kulkarni and Dalai, 2006; Haas *et al*., 2006; Marchetti *et al*., 2008).

Table 1.1 Different feedstock for production of biodiesel

Conventional feedstock		Non-conventional feedstock
Mahua	Soybean	Lard
Nile tilapia	Rapeseed	Tallow
Palm	Canola	Poultry fat
poultry	Babassu	Fish oil
Tobacco seed	Brassica carinata	Bacteria
Rubber plant	Brassica napus	Algae
Rice bran	Copra	Fungi
Sesame	Groundnut	Micro algae
Sunflower	Cynara cardunculus	Tarpenes
Barley	Cottonseed	Latexes
Coconut	Pumpkin	Microalgae
Corn	Jojoba oil	Pongamina pin nata
Used cooking oil	Camelina	Palanga
linseed	Peanut	Jatropha curcas
Mustard	Olive	Sea mango

Biodiesel has a significant influences in reduction of engine emission is such as unburned hydrocarbons (68%), particulars (40%), carbon monoxide (44%), sulfur oxide (100%), and polycyclic aromatic hydrocarbons (PAH) (80-90 %) (Tyson and McCormic, 2006). Meanwhile, it is safer to store and handle and it can be easily produced in domestic quantities.

The investigation of vegetable oils as fuel started in 1978 and 1981 in the United State and South Africa respectively. In 1982, methyl ester was produced in Germany and Austria from rapeseed oil and a small pilot plant was built in Austria at 1985. Commercial production of methyl ester first began in Europe in 1990. The annual production of biodiesel fuel in Europe was 1,210,000 tons in year 2000 and world vegetable oil extraction increased from 56 million tons in 1990 to 88 million tons in 2000 (Demirbas, 2008a).

1.2 Problem statement

The biodiesel production cost highly dependent to the raw material price. Some vegetable oil such as soybean, rapeseed and sunflower are the major feedstocks for biodiesel production but the product is high price biodiesel definitely. The usage of waste cooking oil in this research is a key component for reducing the cost of biodiesel production around 60 % to 90 %.

Many researchers have used alkali catalysts (NaOH, KOH, CH_3ONa) for production of biodiesel because they are cheap and readily available. However, this method has some limitations such as high energy consumption which in turn causes a dramatic increase in capital equipment costs and safety issues. In addition, this process is highly sensitive to water and free fatty acid (FFA) content in the feed stock. This is because high water content can change the reaction to saponification which causing reductions of ester yield, difficult separation of glycerol from methyl ester, increase in the viscosity, and the formation of emulsion (Liu, 1994; Basu and Norris, 1996). Therefore, the heterogeneous solid catalyst is a key component for covering all these problems by simultaneous transesterification and esterification reactions.

There is not any kinetic report for application of heteropoly acid as a catalyst for biodiesel production from WCO. Most importantly, the applications of heterogeneous catalysts have been reported by a lot of researchers recently. Therefore, the kinetic parameters are key factor for simulation and particularly economical industrialization of biodiesel production.

Costly lubrication equipment and time consuming tests are necessary to conduct the optimization of the parameters involved in the transesterification reaction. Therefore, it is of interest to implement some type of simulation and modeling process to accurately predict the biodiesel conversion or yield evolution for various initial conditions of reaction parameters. Thus, software's such as Response Surface Methodology (RSM) and Artificial Neural Network (ANN) can reduce or eliminate a large number of laboratory tests and associated costs.

1.3 Objectives

1) To produce biodiesel from waste cooking oil at the presence of heterogeneous solid acid catalyst.
2) To characterize the biodiesel chemical and physical properties according to ASTM D6751 method.
3) To optimize and model the biodiesel production by response surface methodology (RSM) and artificial neural network (ANN).
4) To find kinetic parameters for transesterification reaction of biodiesel production by experimental method.

1.4 Scopes

In this study, Biodiesel produced from WCO at the presence of tungstophosphoric acid (TPA) as catalyst in a batch-type reactor. Various reaction parameters which include of molar ratio of alcohol to oil, reaction temperature, reaction time, and catalyst concentration investigated to obtain the optimum conditions.

The final product was characterized by ASTM D4052, ASTM D445, ASTM D97, ASTM D2500, ASTM D93, and ASTM D2709 methods to comprise the physical and chemical properties of biodiesel by ASTM D6751. In addition, the researcher used Response Surface Methodology (RSM) and Artificial Neural Network (ANN) for optimization and modeling of transesterification reaction process. Meanwhile, researcher found the kinetic parameters (reaction rate constant (k), activation energy (Ea), and pre-exponential factor (A)) by experimental method at optimum reaction condition.

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