RICE HUSK ASH AS HETEROGENEOUS CATALYST SUPPORT FOR CONVERSION OF WASTE COOKING OIL TO BIODIESEL

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DECLARATION

I declare that this thesis entitled “Rice Husk Ash As Heterogeneous Catalyst Support For Conversion Of Waste Cooking Oil To Biodiesel” is the result of my own research except as cited in the references

Signature	:	*Yohen*
Name	:	Yohen A/L Ganasegaran
Date	:	20/01/2023

APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honours.

Signature :

Supervisor Name : Dr.Mahanum binti Mohd Zamberi

Date : 20/01/2023

DEDICATION

Towards my family and friends.

ABSTRACT

Global fuel demand is rising as a result of rising populations, expanding transportation networks, expanding agricultural, and expanding industry. Renewable resources such as wind and solar power are being used instead of fossil fuels. This research was caried out to crete biodiesel through transesterification process using conventional method by edible waste cooking oil (WCO) as the main feedstock. Acid esterification was conducted by using sulphuric acid (H_2SO_4), to lower the high free fatty acid level of *WCO* and carried on with transesterification process by using batch conventional method. Silica dioxide (SiO_2), obtained from the rice husks were used as solid heterogeneous catalyst together with methanol (MeOH) as the main alcohol used in the transesterification process. The catalyst was calcined at a particular range of 800 ˚C to 1000 °C and was evaluated using X-ray fluorescence (XRF) and scanning electron microscopy (SEM-EDS) techniques. The acid value of the oil was successfully reduced from 4.26 mgKOH/g to 0.337 mgKOH/g. The optimum yield for the fatty acid methyl ester (FAME) reached up to 95.05% with an optimum condition of 12:1 methanol to oil molar ratio, catalyst concentration of 0.5 wt%, and 2 hours of reaction time. By using ASTM D6751 and EN 14214 standards, the quality of the methyl ester produced was evaluated and were found within the standards.

ABSTRAK

Permintaan bahan api global meningkat hasil daripada peningkatan populasi, meluaskan rangkaian pengangkutan, mengembangkan pertanian dan mengembangkan industri. Sumber tenaga yang boleh diperbaharui seperti angin dan tenaga suria digunakan selain daripada bahan fosil. Kajian eksperimen ini dijalankan untuk menghasilkan biodiesel melalui proses transesterifikasi menggunakan kaedah konvensional dengan minyak masak terpakai boleh dimakan (WCO) sebagai bahan mentah utama. Pengesteran asid dijalankan dengan menggunakan asid sulfurik (H_2SO_4), untuk mengurangkan tahap asid lemak bebas *WCO* yang tinggi dan diteruskan dengan proses transesterifikasi menggunakan kaedah konvensional kelompok. Silika dioksida (SiO_2), yang diperoleh daripada sekam padi digunakan sebagai pemangkin heterogen pepejal bersama metanol (MeOH) sebagai alkohol utama yang digunakan dalam proses transesterifikasi. Pemangkin telah dikalsinkan pada julat suhu tertentu 800 °C hingga 1000 °C dan dinilai menggunakan teknik pendarkilau sinar-X (XRF) dan mikroskop imbasan elektron (SEM-EDS). Nilai asid minyak berjaya dikurangkan daripada 4.26 mgKOH/g kepada 0.337 mgKOH/g. Hasil optimum untuk asid lemak metil ester (FAME) mencapai sehingga 95.05% dengan keadaan optimum 12:1 nisbah molar metanol kepada minyak, kepekatan pemangkin 0.5 wt%, dan 2 jam masa tindak balas. Dengan menggunakan piawaian ASTM D6751 dan EN 14214, kualiti metil ester yang dihasilkan telah dinilai dan memenuhi piawaian yang ditetapkan.

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LIST OF SYMBOLS AND ABBREVIATIONS

WCO	-	Waste cooking oil
SiO_2	-	Silica oxide
FAME	-	Fatty Acid Methyl Esters
RH	-	Rice Husk
EDS	-	Energy Dispersive Spectroscopy
H_2SO_4	-	Sulphuric Acid
ULSD	-	Ultra Low Sulphur Diesel
CO_2	-	Carbon Dioxide
SO_2	-	Sulphur Dioxide
SEM	-	Scanning Electron Microscopy
EN	-	European Norn
MeOH	-	Methanol
FFA	-	Free Fatty Acid
NOx	-	Nitrogen Oxide
KOH	-	Potassium Hydroxide
NaOH	-	Sodium Hydroxide
RHA-Ash	-	Rice Husk Ash
Mgo	-	Magnesium Oxide
CaO	-	Calcium Oxide
TAG	-	Triacylglycerol
XRF	-	X-Ray Fluorescence
GC-MS	-	Gas Chromatography-Mass Spectrometry
%wt	-	Weight percentage

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CHAPTER 1

INTRODUCTION

1.1 Background

Energy demand rises in lockstep with population growth, industrialization and transportation requirements. Natural gas, coal, and petroleum are all fossil fuels that will eventually run out of energy. Rapid urbanization, combined with higher usage of fossil fuels, has elevated the environment globally. Thus, dwindling global reserves of fossil fuels and increased awareness of the public towards environmental pollution have raised a demand for renewable, non-toxic, and efficient fuels. Burning conventional fossil fuels emits harmful gases such as sulfur oxides, carbon dioxides, nitrogen oxides, and other air pollutants which have numerous consequences for human health and the environment.

There are many researchers who introduced clean and user-friendly fuels in response to the aforementioned issues. According to (Ambat et al., 2018), biodiesel is a renewable fuel that has excellent properties that make it compatible with nature and the environment, making it a promising alternative fuel. Biodiesel has been a taboo topic for a long time as it is substitutional for non-renewable energy that has various positive characteristics such as being eco-friendly and containing renewable behavior. Mono-alkyl esters of animal fats or vegetables are actually biodiesel. Biodiesel has various advantages as it is biodegradable.

Global warming leads to incurable disease and climate change as well, can be countered by a certain percentage by using biodiesel as it can decrease the emission of sulphur oxide and reduces greenhouse effects. In order to produce biodiesel as renewable fuel animal fats, vegetable oils, as well as micro- algae, have to undergo chemical processes. This process is known as the reaction between crude oil with various kinds of catalysts and alcohols.

After adding all these substances together, it is then purified to a better form. Once it is purified, it will be tested to obtain yields that are sufficient and compatible enough to run in a diesel engine. This biodiesel can be used in a pure diesel engine or it can be blended with diesel fuel according to a certain ratio for any diesel engine without specification or modifications. Using this biodiesel, modifications to diesel engines are not necessary. Moreover, it is safer to use and this biodiesel would have the quality and specification according to the American Society Of Testing and Materials (ASTM D6751).

In the year 1853, the transesterification of vegetable oil was carried out by Patrick Duffy years before the first diesel engine was in use. Decades later, Rudolf Diesel created an iron cylinder in a size of 10 ft (3.05 m) together with a flywheel base (Razzaq et al.,2021). It was the first biodiesel-powered vehicle which was Rudolf Diesel's prime model that ran on this fuel in Ausburg, Germany in the year 1893. To be specific, that vehicle was only powered by peanut oil. As time passes by, the lack of quality of the fuel spray caused by the thickness (viscosity) of the vegetable oil caused damage to the engines.

Therefore, scientists managed to then conduct experiments to improvise vegetable oil into biodiesel in the year 1984 by using the transesterification method. Another reason why scientists needed to find an alternative is that they wanted a lower viscosity oil in order to combust it throughout the diesel engine. Thus, they managed to convert vegetable oils into fatty acid alkyl esters and used them as a substitute for diesel. This diesel that was produced was less viscous and had a better combustion rate.

1.2 Problem Statement

The drastic advancement of our population will head toward higher energy demand globally in the long term run. The major usage of energy of all countries is pointing towards petrochemical resources which are also non-renewable energy that is limited. An increase in demand in this industrialized world will only lean against excessive usage of these petroleum derivatives. Thus biodiesel is a substitute way to replace diesel petroleum oil usage as it is more environment-friendly, biodegradable, and safer to use.

The only setback to obtain biodiesel would be the cost to conduct the whole process to produce biodiesel. The price of alcohol and crude oil is relatively expensive. Besides that, measures and variables such as the methanol to oil molar ratio and concentration of catalyst, and reaction time must be taken down. Frequent tests should be conducted until the oil turns into proper biodiesel referring to the ASTM D6751 standards. So, time, energy and money play a major role in the production of biodiesel.

Thus, this research was conducted not only to obtain a good biodiesel using raw materials but to also reduce the cost, energy and time to product it. Two most important raw materials that can be reused will be used to obtain biodiesel which are rice husks and waste cooking oil (WCO). The reason why rice husks were selected is because it is available abundantly and cheap.

Moreover, rice husks are not widely used for various purposes as it only causes environmental issues because it is not decomposed properly at the paddy field but it is found that rice husks have high content of silica which will be useful to produce a good heterogeneous catalyst.

By using heterogeneous catalyst, washing processes will not be necessary and time can be reduced to make biodiesel. Besides that, waste cooking oil (WCO) that are not well disposed leads to pollution and causes harm to aquatic life. *WCO* is also available almost everywhere in the country and it is also not expensive. In a nutshell, it can be concluded that using *WCO* and rice husks will be good raw materials to produce biodiesel.

1.3 Objective

1. To produce and characterize the high catalytic catalyst from waste rice husk.
2. To determine the variables, effects such as reaction time, reaction temperature, catalyst loading and methanol to oil molar ratio on the biodiesel production.

1.4 Scope

1. To check fuel properties of raw feedstock (WCO).
2. Producing the heterogeneous catalyst by calcination process ranging from 800 °C to 1000 °C.
3. To understand and observe catalyst characterization (SEM, XRF).
4. Conduct biodiesel production via conventional batch transesterification.
5. Undergo GC-MS analysis to check the quality of the production of B100.
6. To achieve fuel properties according to ASTM D6751 and EN 14214.

CHAPTER 2

LITERATURE REVIEW

2.1 Background of Biodiesel

Triacylglycerol (TAG) are the primary constituents of both plant and animal lipids. Multiple fatty acids are commonly found in animal fats and vegetable oil. The total fatty acid of the animal fat and vegetable oil is based on the total content of the fatty acid of the *TAG*. In determining the qualities of animal fats or vegetable oil, the fatty acid profile is arguably the most critical characteristic that influences the correlating properties of a vegetable oil or animal fat.

This is due to different fatty acids have different physical and chemical qualities because each fatty acid has unique physical and chemical properties. In order to produce biodiesel, animal fat and vegetable oil would undergo a process called transesterification. The animal fat and vegetable oil react along with the presence of the catalyst and alcohol to produce alkyl esters of the fatty acid mixture that is present in the animal fat or vegetable oil. Production of biodiesel can be carried out with various feedstock.

Some of the examples of vegetable oils are (palm oil, sunflower oil, coconut oil, peanut oil, and soybean) whereas for animal fats, it commonly frying oil or waste cooking oil. Changes in the production process may be necessary depending on the quality of the feedstock. 100% biodiesel is known as pure biodiesel or B100 (Leite et al., 2019). Pure biodiesel blended with petrodiesel is actually biodiesel blend. Normally, biodiesel blends are written as *BXX*. The *XX* represents the volume of biodiesel in the blend. For instance, a B70 is 70% biodiesel and 30% petrodiesel.

The alcohol used to produce biodiesel is methanol as it is lower in cost but alcohols such as iso-propanol and ethanol may lead to a better production of biodiesel with quality properties (Katam & Bhattacharyya, 2018). Fatty acid methyl esters (FAME) are also another name for biodiesel that will be used once the resulting products are obtained.

2.1.1 Production of Biodiesel

The transesterification consists of fats and oils reactions on *TAG* approximately 15 to 23 atoms with a low molecular weight of alcohol (generally methanol or ethanol) along with a base catalyst to deliver glycerine and esters. Normally, reaction occurs at an atmospheric pressure around 60 °C to 65 °C. The process takes up around two and half hour of time to conduct and kept for filtering for 24 hours.

The transesterification contains three reversible and continuous reactions Figure 2.1. Three moles of alcohol and one mole of triglyceride is the stoichiometric ratio for the transesterification reaction. In order to move the reaction to the methyl esters formation, an additional measure of alcohol us included.

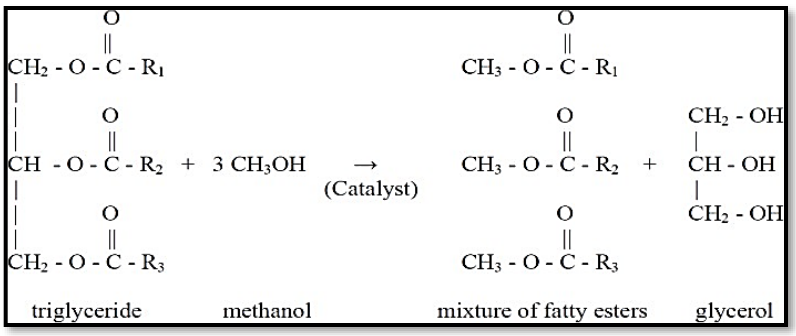

Figure 2.1: Transesterification chemical reactions (Chozhavendhan et al. 2020)

2.1.2 Properties of Biodiesel

In contrast to low-sulfur diesel fuels, biodiesel has the potential to have beneficial lubricating characteristics and higher cetane ratings. Fuels with increased lubricity may lengthen the useable existence of high-pressure fuel injection hardware since this hardware receives its lubrication from the fuel itself. This may include high pressure injection pumps, pump injectors, and fuel injectors, but it all depends on the engine.

The feedstock that is used has a far greater impact on the energy density of the resulting biodiesel than the manufacturing method does. According to (Cloin, 2006) it has been argued that biodiesel delivers superior lubricity and increasingly complete combustion, in this way widening the engine energy output and somewhat making up for the higher energy density of petrol-based diesel fuel.

Depending on the process used to produce it, biodiesel may seem anywhere from a light golden colour to a dark brown hue. It has a high boiling point, but its vapour pressure is modest, and it is only slightly miscible with water. Since the sulphur mixtures in petroleum diesel are responsible for a significant portion of the lubricity, biodiesel is typically utilised as an added substance to ultra-low sulphur diesel (ULSD) (Dhahad et al., 2019) fuel to assist with lubrication because biodiesel does not contain any sulphur.

2.1.3 Engine Performance

The amount of power that can be produced by biodiesel is dependent on its mix, its quality, and the load conditions that are present when the fuel is burned. Because various blends include different kinds of energy, their thermal efficiencies will be different from one another. For example, the thermal efficiency of B100 will be different from that of B20. These qualities will alter as the mixes just as the quality of biodiesel fluctuates. The thermal