



**CATALYTIC HYDROPROCESSING OF FATTY ACIDS TO RENEWABLE
DIESEL USING LANTHANUM-MODIFIED ZEOLITE-BASED CATALYSTS**

By

NUR AZREENA BINTI IDRIS

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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DEDICATION

This thesis is dedicated to my late father Idris Bahrudin, mama Azizah Abdul Halim, mak Jamilah Abdul Rahman, ayah Zainal Abidin Ahmad and husband Mohd Hafirul Zainal Abidin

My beloved son, Muhammad Fateh Aydeen Mohd Hafirul

With love, respect and a bunch of memories

Indeed, we belong to Allah and indeed to Him we will return.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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Faculty : Science

The hydroprocessing of fatty acids into hydrocarbons is a viable alternative approach for producing renewable fuel with improved properties than biodiesel, which has inherent drawbacks due to its high oxygen content. Therefore, this study explored the catalytic hydroprocessing of fatty acids into renewable diesel using zeolite based-catalysts (zeolite beta and HZSM5). The optimal conditions established for both catalysts were 350 °C, 4 MPa hydrogen pressure, and 5 wt.% of catalyst loading for 2 h. Zeolite beta outperformed the HZSM5 in terms of HDO reaction activity, with a diesel selectivity of 77% compared to HZSM5 (70%). Furthermore, the La₍₁₀₎ZE0(90) catalyst demonstrated conversion of OA up to 99% with 83% of C₁₅ and C₁₇ selectivity. The superior activity of La₍₁₀₎ZE0(90) was attributed to the synergistic interaction of La-Si-Al, a sufficient number of weak and medium acid sites, and excellent textural properties of the catalyst. Interestingly, when La₍₁₀₎ZE0(90) was applied to palm fatty acid distillate (PFAD) for the renewable diesel production, catalytic deoxygenation (DO) and HDO reactions were involved. The DO reaction was carried out at 350 °C in a nitrogen environment with 5 wt.% catalyst loading for 3 h reaction time, whereas the HDO reaction was carried at 400 °C and 5 MPa, respectively. Remarkably, the HDO reaction of PFAD produced renewable diesel (RD 100) containing 73% of C₁₆ selectivity, whereas the DO reaction produced 51% of C₁₅ via the deCO_x route. The RD 100 produced via the HDO and DO reactions met ultra-low sulphur diesel (ULSD) specifications, indicating that it can be used directly in automobile engines or blended with conventional diesel to significantly improve the fuel characteristics profile. Meanwhile, the performance of La₍₁₀₎HZSM5(90) was evaluated using used frying oil (UFO) and PFAD, using 5 wt.% catalyst loading, 2 h reaction time, 5 MPa H₂ pressure, and 400 °C. The diesel yields were 85% (UFO) and 93% (PFAD) respectively. The UFO showed a selectivity of 19% for C₁₇ fraction and 51% for C₂₀ fraction while PFAD exhibited C₁₅ (48%) and C₂₀ (29%). Additionally, the La₍₁₀₎HZSM5(90) catalyst exhibited a promising cycle with five consecutive runs despite coke formation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**HIDROPEMROSESAN BERMANGKIN ASID LELEMAK KEPADA DIESEL
BOLEH DIPERBAHARUI MENGGUNAKAN MANGKIN BERASASKAN
ZEOLIT DIUBAHSUAI LANTHANUM**

Oleh

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Hidropemprosesan asid lemak kepada hidrokarbon adalah pendekatan alternatif untuk menghasilkan bahan api boleh diperbaharui yang mempunyai pencirian yang lebih baik daripada biodiesel yang mempunyai kelemahan akibat kandungan oksigen yang tinggi. Oleh itu, kajian ini meneroka hidropemprosesan bermangkin asid lemak kepada diesel boleh diperbaharui menggunakan pemangkin berasaskan zeolit (zeolit beta dan HZSM5). Keadaan optimum bagi kedua-dua pemangkin ialah 350 °C, tekanan hidrogen 4 MPa, dan 5 wt.% mangkin selama 2 jam. Zeolit beta menunjukkan aktiviti tindak balas HDO yang lebih baik daripada HZSM5 dengan selektiviti diesel sebanyak 77% berbanding HZSM5 (70%). Oleh itu, pemangkin berasaskan zeolit beta yang diubah suai logam turut dikaji kepada OA pada suhu 350 °C selama 2 jam di bawah tekanan 4 MPa H₂. La₍₁₀₎zeo₍₉₀₎ menunjukkan penukaran OA sebanyak 99% dengan 83% selektiviti C₁₅ dan C₁₇. Prestasi yang baik bagi La₍₁₀₎zeo₍₉₀₎ adalah disebabkan oleh interaksi sinergistik La-Si-Al, bilangan tapak asid lemah dan sederhana yang mencukupi, dan sifat tekstur pemangkin yang sangat baik. Menariknya, apabila La₍₁₀₎zeo₍₉₀₎ digunakan pada asid lemak sawit tersuling (PFAD) untuk menghasilkan diesel boleh diperbaharui, dua tindakbalas terlibat iaitu penyahoksigenan bermangkin (DO) dan HDO. Tindak balas DO telah dijalankan pada 350 °C dalam persekitaran nitrogen, 5 wt.% mangkin selama 3 jam, manakala tindak balas HDO dijalankan pada 400 °C dan 5 MPa tekanan H₂ selama 3 jam. Tindak balas HDO menghasilkan diesel boleh diperbaharui (RD 100) yang mengandungi 73% selektiviti C₁₆, manakala tindak balas DO menghasilkan 51% C₁₅ melalui laluan penyahkarboksilan. RD 100 yang dihasilkan melalui tindak balas HDO dan DO memenuhi spesifikasi diesel ultra-rendah sulfur (ULSD), menunjukkan bahawa ia boleh digunakan terus dalam enjin kereta atau dicampur dengan diesel konvensional untuk meningkatkan profil ciri bahan api. Sementara itu, aktiviti La₍₁₀₎HZSM5₍₉₀₎ dikaji menggunakan minyak goreng terpakai (UFO) dan PFAD, pada suhu 400 °C, tekanan H₂ 5 MPa,

5 wt.% La₍₁₀₎HZSM5₍₉₀₎ selama 2 jam dengan 85% diesel terhasil dari UFO dan 93% dari PFAD telah diperolehi. UFO menunjukkan selektiviti 19% untuk C₁₇ dan 51% untuk C₂₀ manakala PFAD menunjukkan selektiviti C₁₅ (48%) dan C₂₀ (29%). Selain itu, pemangkin La₍₁₀₎HZSM5₍₉₀₎ menunjukkan kebolegunaan semula mangkin sebanyak lima kali walaupun terdapat pembentukan kok.



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LIST OF ABBREVIATIONS

GHG	Greenhouse gas
SDG	Sustainable development goal
FAME	Fatty acid methyl ester
DO	Deoxygenation
CFPP	Cold filter plugging point
DCX	Decarboxylation
DCN	Decarbonylation
HDO	Hydrodeoxygenation
BTL	Biomass to liquid
PFAD	Palm fatty acid distillate
MPOB	Malaysian Palm Oil Board
FOB	Free On Board
UFO	Used frying oil
EU	European union
RE	Rare earth
OA	Oleic acid
HZSM5	Zeolite Socony Mobil-5
XRD	X-ray diffraction
SEM	Scanning electron microscope
TPD-NH ₃	Ammonia-Temperature Programmed Desorption
TGA	Thermogravimetric analysis
BET	Brunauer-Emmett-Teller
CNHS	Carbon, Hydrogen, Nitrogen, Sulphur elemental analysis
OVAT	One Variable-at-a Time
CLP	Crude liquid product

RLP	Refined liquid product
GC-FID	Gas chromatography-flame ionization
GCMS	Gas chromatography-mass spectrometry
GC-TCD	Gas chromatography-thermal conductivity detector
FT	Fisher-Tropsch
FFA	Free fatty acid
HEFA	Hydroprocessed esters and fatty acids
BHD	Bio-hydrogenated diesel
HDRD	Hydrogenation derived renewable diesel
CN	Cetane number
WHSV	Weight hourly space velocity
LSHV	Liquid space hour velocity
HDS	Hydrodesulphurization
LCO	Light cycle oil
SEA	Strong Electrostatic Adsorption
IA	Ion Adsorption
EDF	Equilibrium Deposition Filtration
HDN	Hydrodenitrogenation
HDM	Hydrodemetallization
TAN	Total acid number
FAC	Fatty acid composition
FTIR	Fourier Transform Infrared
KOH	Potassium hydroxide
BJH	Barret-Joyner-Halenda
EDX	Energy dispersive X-ray analyzer
TMO	Transition metal oxide
AOCS	American Oil Chemists' Society

NIST	National institute of standards and testing
MOFs	Metal organic frameworks
ULSD	Ultra-low sulphur diesel
CO ₂	Carbon dioxide
JCPD	International Centre for Diffraction Data
PSD	Pore size distributions
RPS	Refined palm stearin
RPO	Refined palm olein
Nox	Nitrous oxide
H ₂ S	Hydrogen sulphide
NZ	Natural zeolite
SAPO-11	Silicoaluminophosphates
HDA	Hexadecylamine
OxA	Oxalic acid
Pd	Palladium
Pt	Platinum
Rh	Rhodium
Ce	Cerium
La	Lanthanum
La ₂ O ₃	Lanthanum oxide
CeO ₂	Cerium oxide
Nd ₂ O ₃	Neodymium oxide
Pr ₆ O ₇	Praseodymium oxide
FCC	Fluid catalytic cracking
SOFCs	Solid oxide fuel cells
CO	Carbon monoxide
HDT	Hydrotreating

HDC	Hydrocracking
FCC	Fluid catalytic cracking
HVOs	Hydrotreated vegetable oils
OA	Oleic acid
CO	Carbon monoxide
NaOH	Sodium hydroxide
HC	Hydrocarbon
APR	Aqueous phase reforming
DeCO _x	Decarboxylation/decarbonylation
CN	Cetane number
CI	Cetane index
H ₂	Hydrogen
HDN	Hydrodenitrogenation
HY	H-type Y zeolite
FID	Flame ionization detector

CHAPTER 1

INTRODUCTION

1.1 Research background

Approximately 20% of the world's energy requirements are met by transportation fuels, which are derived from fossil fuels (Shi *et al.*, 2017). The drastic increase in the number of automobiles, advanced industrialisation, rapid urbanisation has caused the depletion of fossil fuels and increased environmental awareness (Why *et al.*, 2019). The combustion of fossil fuels is a key factor that contributes to the spike in Greenhouse Gas (GHG) emissions and climate change (Wang *et al.*, 2020). Past studies have conveyed that climate change mitigation was consistent with the Sustainable Development Goal 13 (SDG 13; climate action), which is deemed the most challenging issue that affect both developed and developing nations (Torres *et al.*, 2021). One of the most feasible approaches to combat climate change is to maximise the utilisation of renewable energy sources (SDG 7: Affordable and Clean Energy) (Purnomo *et al.*, 2018).

Renewable energy technologies, such as solar, wind, hydrogen, fuel cells, and biomass, are considered promising alternatives to meet the future global energy demand (Eagan *et al.*, 2017). Currently, numerous renewable energy research has focused on extracting energy from biomass sources, which can provide solid, gaseous, and liquid fuels with the greatest potential to cater to future global energy demands (Liu *et al.*, 2018). Biomass is also an attractive renewable source for biofuel production given its naturally abundant, economically viable, and ecologically friendly features (Baharudin, Taufiq-Yap, *et al.*, 2019). The use of biofuels derived from renewable resources, such as biomass and vegetable oils, could serve as an alternative fuel source to reduce the overdependency on fossil fuels and minimise the GHG emission (Douvartzides *et al.*, 2019). Therefore, there is an urgent need for the development of renewable energy sources that can effectively replace fossil fuels (Yu *et al.*, 2013). In this regard, biofuel has been suggested as a replacement for fossil fuel.

Biofuel is defined as solid, liquid, or gaseous fuels derived primarily from bio-renewable feedstocks (Fatih Demirbas, 2009). It can be categorised as first-, second-, or third-generation based on the applied production technologies (Hongloi, Prapainainar and Prapainainar, 2021) as well as the type of biomass it utilises (Alalwan, Alminshid and Aljaafari, 2019). For instance, food crop feedstocks, primarily corn and sugarcane, are the source of first-generation biofuels, which include bioethanol, biogas, and biodiesel (fatty acid methyl esters, or FAME), while second-generation biofuels are basically hydrocarbon fuels produced by catalytic deoxygenation (DO) or the Fischer-Tropsch process from edible and non-edible feedstocks (Di Vito Nolfi, Gallucci and Rossi, 2021). Meanwhile, Brandão *et al.*, 2021 stated that third-generation biofuels are generated using cyanobacteria, yeast, fungus, and algae.

Biodiesel, the first-generation biofuel, is produced through a transesterification process in which vegetable oil or animal fat (triglyceride) reacts with alcohol in the presence or absence of a catalyst to produce the corresponding alkyl esters of the fatty acid mixture found in the parent vegetable oil or animal fat (Kombe *et al.*, 2013). However, some concerns have emerged regarding the use of biodiesel as a result of its practical applications as an alternative to petroleum diesel. These issues include a shorter shelf life and inferior cold flow properties, such as lower cold-temperature fluidity and cold filter plugging point (CFPP), as well as engine compatibility (Wang *et al.*, 2018). These issues are primarily caused by the high oxygen content in its composition (Lee *et al.*, 2021). Consequently, in recent years, several approaches have been proposed and developed to remediate these problems (Hachemi *et al.*, 2017).

Hydroprocessing, which involves selective catalytic cracking or DO via decarbonylation (DCN), decarboxylation (DCX), or hydrodeoxygenation (HDO) processes, is used to convert vegetable oils and fatty acids as well as lipids from microalgae into fuels such as conventional diesel (Plaola *et al.*, 2022). The majority of reactions emphasise the importance of multiple operating parameters, such as the type of supporting metal catalyst, the type and rate (residence time) of feed, reaction temperature, reaction atmosphere, H₂ partial pressure, amount of catalyst, and type of solvent, in determining the overall yield and selectivity of the diesel-like hydrocarbon fuels (Yao *et al.*, 2021).

Recently, attention has been drawn to technologies such as biomass to liquid fuels (BTL) and hydrotreatment of vegetable oils to produce renewable diesel. The biofuel growth trend in biofuels industry is depicted in Figure 1.1 (Arun, Sharma and Dalai, 2015). Although vegetable oils are commonly converted into transportation fuels (Mansir *et al.*, 2017), the high viscosity of crude vegetable oil (often in the range of 28 - 40 mm² s⁻¹) leads to operational issues in diesel engines, such as deposit formation and injector coking due to poorer atomisation upon injection into the combustion chamber, making it an unfavourable option to produce biofuel (Ashraf Amin, 2019). Therefore, bio-based sources, specifically fatty acids, are sustainable resources with high energy densities that can be efficiently converted into liquid fuel (Bjelić *et al.*, 2018).

Palm fatty acid distillate (PFAD) is an example of a readily available fatty acids by-product from the refinement of crude palm oil in the oil palm industry. According to the Malaysian Palm Oil Board (MPOB), every 100 tonnes of refined palm oil produces approximately 4.8 tonnes of PFAD, and a plentiful supply of 622,309 tonnes of PFAD was generated in 2021 with a Free On Board (FOB) price of RM 4,233 per tonne (Malaysian Palm Oil Board, 2021). Contrarily, used frying oil (UFO) is the by-product generated from frying food in cooking oil, which contains refined plant- or animal-based fats (Lycourghiotis *et al.*, 2019). According to previous reports, UFOs are generated in large quantities in the European Union (EU) between 700,000 and 1,000,000 tonnes annually, while Asian nations, such as China, Malaysia, Indonesia, Thailand, Hong Kong, and India, generated around 40,000 tonnes of UFOs annually (Alsultan *et al.*, 2021).

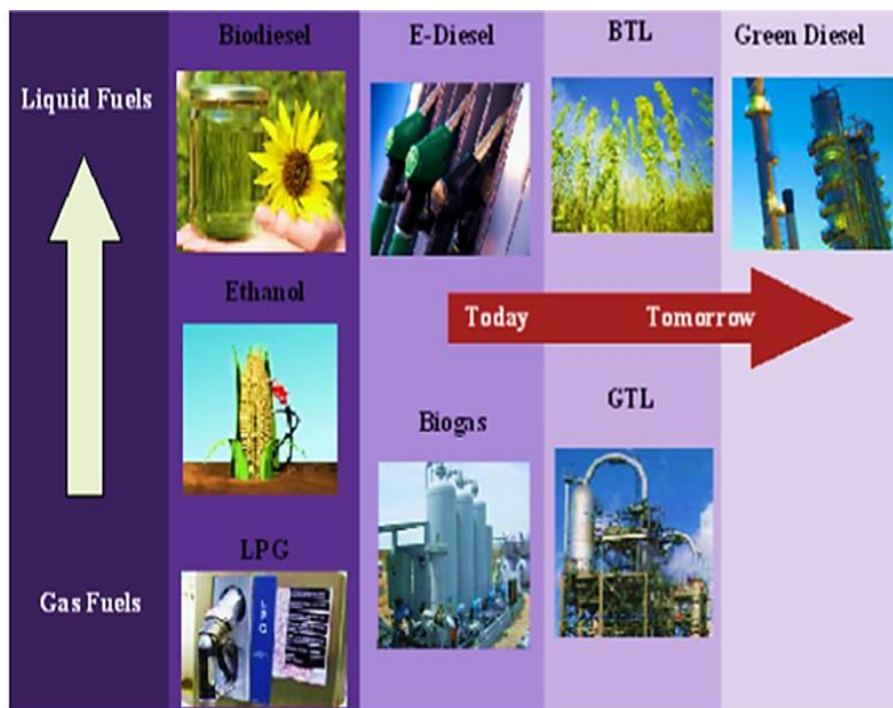


Figure 1.1 : The biofuel growth trend (Arun, Sharma and Dalai, 2015)

In addition, catalysts play a vital role to enhance the rate of hydroprocessing while excellerate the fatty acid conversion from fatty acids into biofuel. For example, the conversion of unsaturated molecules into saturated compounds and the removal of heteroatoms (Taromi and Kaliaguine, 2018). Noble-metal catalysts (Pt, Pd, etc.) and sulfided catalysts (Co-Mo, Ni-Mo) were traditionally used for hydroprocessing (primarily HDO). Nevertheless, the use of noble metals substantially raises the price of catalysts, whereas sulfided catalysts necessitate the addition of sulphur-containing agents in order to maintain their stable activity. The use of sulfided catalysts frequently results in sulphur pollution during the production of fuels, which is a significant disadvantage. Transition metals such as nickel (Ni), molybdenum (Mo), zinc (Zn), and iron (Fe) were also used as active components, however, they are prone to coke formation (Nuhma *et al.*, 2022).

Meanwhile, rare earth (RE) elements are essential components of the commercial catalysts for the catalytic cracking of hydrocarbons (Xiaoning *et al.*, 2007). The REs have recently received much attention regarding energy-efficient petroleum cracking from refined crude oil into gasoline, distillates, lighter oil products, and other fuels. They are also responsible to remove leaded gasoline (Akah, 2017). Lanthanum oxide has already been studied in catalysis as a support for Ni catalysts in ethanol steam reforming (Lanre *et al.*, 2020); as a catalyst in methane reforming (Kalai *et al.*, 2017) and as a catalyst for hydrogen

production from steam reforming or oxidative steam reforming of ethanol (de Lima *et al.*, 2010) and for biodiesel production (Russbuedt and Hoelderich, 2010; Vieira *et al.*, 2013; Rattanaphra *et al.*, 2019),

Catalysts with high metal loading, exhibit poor catalytic performance during chemical removal of O atoms from fatty acid (Peng *et al.*, 2013). In this regard, supports are at least as important as the metal itself to the distribution and decrease of metal particles (Dusescu *et al.*, 2018). Zeolites have also been extensively applied as solid acid catalysts or catalyst carriers in the oil refining and petrochemical industries, which are gradually replacing conventional homogeneous and heterogeneous catalysts (de Oliveira Camargo *et al.*, 2020). Zeolites exhibit various unique properties that make them suitable to catalyse organic chemical processes, including their crystalline structure with porous and large surface areas, homogenous microporosity, high hydrophobicity, shape selectivity, acid-base properties, and high resistance to deactivation by carbon deposition (Aziz *et al.*, 2020). The low cost of production and biodegradability features also allow zeolites to be widely applied in numerous industrial heterogeneous catalytic applications. Furthermore, the molecular pores of zeolites can readily absorb smaller molecules while excluding larger molecules, thus acting as molecular sieves (Yan *et al.*, 2021). This characteristic also permits zeolite to function as an ion exchange media. For instance, Al^{+3} can be used in place of Si^{+4} inside the framework of crystalline silica (SiO_2), which generates negative charges inside the framework of the catalyst and increases its catalytic activity (Shahinuzzaman, Yaakob and Ahmed, 2017).

1.2 Problem statements

Demand for fossil fuels is rising as a result of increased global industrialization and motorization (Mandal and Cho, 2022). However, the massive use of fossil fuels to meet the energy needs of today's society has a severe influence on the environment due to the high levels of hazardous gases like NO_x and SO_x , photochemical oxidants, lead compounds, and particulate matter in the atmosphere, (Díaz-Pérez and Serrano-Ruiz, 2020). Moreover, due to high demand, the fossil fuel sources are being depleted, and caused an increase in its prices and makes the substitution of fossil fuel with biofuel more vital (Martinez-Hernandez *et al.*, 2019).

The production of biofuel has played an important role in the global economy and environmental protection. It can be derived from biomass and are considered an eco-friendly, non-toxic, and sustainable alternative to fossil fuels (Janampelli and Darbha, 2018). Traditional biodiesel (FAME) is produced from vegetable oils by homogeneously-catalyzed transesterification with methanol (Mansir *et al.*, 2017). Even though FAME is suitable for blending with petroleum-derived diesel fuels, it suffers from some drawbacks, the main being low oxidation stability that limits its long-term storage and the use of biodiesel–diesel blends containing more than 20 – 30 vol.% of FAME (Manchanda, Tyagi and Sharma, 2018). Therefore, production of renewable diesel through

hydroprocessing that is fully compatible with fossil fuels should be developed. Method to produce renewable diesel that have garnered substantial attention from academics and industry is the catalytic hydroprocessing of liquid biomass, which is utilised to remove heteroatoms (sulphur, nitrogen, oxygen, and metals) as well as for the cracking and saturation of olefins and aromatics (Bjelić *et al.*, 2018). Catalytic HDO is one of the hydroprocessing methods to enhance the physico-chemical and fuel properties of liquid biofuels through the removal of oxygen and moisture content.

The feedstock for renewable diesel production depends on several factors, such as the commercial availability and oil yield from the seed. Raw oil is unsuitable for diesel engines due to its high viscosity and low volatility. In contrast, oleic acid (OA) is usually used as a reactant for renewable diesel production because it is the main component in vegetable oil, such as palm oil, sunflower oil, and jatropha oil (Hongloi *et al.*, 2021). Nevertheless, the use of edible vegetable oils as a material for renewable biofuel production instead as a crucial source of food raises concern over the possible disruption of global food security and food shortage (Tuli and Kasture, 2021). Hence, it is crucial to search for a residual resource, such as PFAD and UFO as it would not only mitigate the excessive production of agricultural by-products but also replace edible oils to produce second-generation renewable diesel production. Besides the low-commodity value, the free fatty acids content of PFAD could be easily converted into sustainable renewable diesel and address the current problems of improper disposal of PFAD into waterways. In addition, the use of PFAD is advantageous because it does not compete with the food industry (Kantama *et al.*, 2015).

Conventional HDO catalysts are predominantly composed of sulphided forms of silica and alumina-supported that results in sulphur residues in the end products and the emission of harmful H₂S (Afshar Taromi and Kaliaguine, 2017). In addition, the use of active supported noble metal catalysts is not advised due to their exorbitant cost, which renders the procedure uneconomical. Moreover, noble metal catalysts are extremely sensitive to toxins and impurities in the feedstock (such as sulphur, heavy metals, and oxygenated compounds) and could lead to a considerable rate of catalyst deactivation (Ameen *et al.*, 2020).

Zeolites, a well-known support catalysts that have been well-utilised in industrial processes, such as catalytic cracking, isomerisation, and alkylation reactions (Foraita *et al.*, 2017) appears to have desired surface area, and pore sizes that are appropriate for hydrocarbon fuel production via HDO reaction (Srihanun *et al.*, 2020). However, the presence of a substantial amount of Bronsted acidity and the cage structure of zeolite provides suitable active sites for coke formation, resulting in the deactivation of the catalyst. In addition, the dealumination process at high temperatures may cause the zeolite structure to collapse, resulting in denser crystalline phases (Simancas *et al.*, 2021).

The addition of RE elements, which frequently display basic characteristics, has been shown to reduce the acidity of zeolite and modifies the basicity of zeolite

catalysts (Sousa-Aguiar *et al.*, 2013). RE elements can sustain catalyst efficacy and boost the product yield by breaking heavier oil fractions. Moreover, La is an environmentally friendly and cheap RE that has been added to catalyst carriers to increase their thermal resistance and surface basicity (Escobar *et al.*, 2019). So, an optimum RE content must be found to meet the output goals. Although the influence of RE element modification on the catalytic performance of zeolite for hydrocarbon cracking has been reported, there are few publications on zeolite-RE catalysts for renewable diesel production.

While the prospect of renewable diesel has become more attractive with numerous reports on the development of zeolite-based catalysts to catalyse the production of renewable diesel, few studies have been published regarding the potential use of La-modified zeolite-based catalysts for renewable diesel production. Therefore, the current work will highlight the effectiveness of the novel La-modified zeolite-based catalyst that are affordable, ecologically acceptable, recyclable and easily functionalised for the production of renewable diesel which indirectly improve the waste management of the oil palm by-product such as PFAD and UFO.

1.3 Research objectives

The purpose of this research is to produce renewable diesel from OA and palm products (UFO and PFAD) catalysed by highly efficient metal modified zeolite-based catalyst. There are three main objectives that have been highlighted:

1. To synthesise, characterise and evaluate the metal modified zeolite-based catalyst performance towards hydroprocessing of OA.
2. To optimise the catalytic HDO reaction at various catalytic parameters and experimental reaction conditions (e.g., different range of temperature, H₂ pressure, amount of catalysts loading, and the variation of reaction time).
3. To apply the optimised catalytic performance of the metal-modified zeolite-based catalyst to palm-based feedstocks and evaluate the renewable diesel characteristics as a potential alternative biofuel.

1.4 Scope of research

This study commenced with the screening of two zeolite-based catalysts, namely zeolite beta and Zeolite Socony Mobil-5 (HZSM) as the supported catalyst, followed by the synthesis of metal-modified zeolite-based catalysts. The metal-modified zeolite beta catalyst was synthesised via the wet impregnation method using various metals, namely La, Co, Fe, Mg, Mn, and Zn. Subsequently, the physical properties of the catalysts were characterised using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Brunauer-Emmet-Teller (BET), Ammonia Temperature-Programmed Desorption (TPD-NH₃), Thermogravimetric

Analysis (TGA), and Carbon, Hydrogen, Nitrogen, and Sulphur elemental analysis (CHNS).

The study was then extended to the evaluation of the HDO reaction of OA as the fatty acid model compound using the metal-modified zeolite beta and HZSM5 catalyst, followed by HDO of OA utilising La-modified zeolite-based catalyst and implementation of the optimal HDO condition for palm-based feedstock. Next, the screening and catalytic performance evaluations were conducted to determine the optimal catalyst for the related catalytic hydroprocessing process. Additionally, the “One Variable-at-a Time” (OVAT) optimisation technique was employed using a variety of parameters, including reaction temperature (300 – 400 °C), hydrogen (H₂) pressure (1 – 5 MPa), catalyst concentration (1 – 7 wt.%), and metal loading (5 – 20 wt.%). This study also focused on the product selectivity, conversion, and hydrocarbon yield of model compounds in the presence of supported catalysts.

The vacuum distillation was used to purify the Crude Liquid Product (CLP) and produce the Refined Liquid Product (RLP). The CLP and RLP were then quantitatively and qualitatively analysed using the Gas Chromatography-Flame Ionisation Detector (GC-FID) and Gas Chromatography-Mass Spectrometer (GC-MS), respectively, to determine the hydrocarbon yield and selectivity of the liquid product whereas the gas products were determined using the Gas Chromatography-Thermal Conductivity Detector (GC-TCD). Finally, the optimised reaction conditions were applied to realistic feedstocks, including UFO and PFAD. The fuel properties were determined using the method specified in the American biodiesel standard (American Society for Testing and Materials, ASTM D6751-2).

1.5 Significance of the study

The use of La-modified zeolite-based catalyst in hydroprocessing of OA was expected to produce renewable diesel. The application of zeolite beta (12-membered ring channels) and HZSM5 (10-membered rings) is appropriate for renewable diesel production due to their large micropores. It was hypothesised that La would function as a highly advantageous acid-base metal promoter for the DCX pathway and also as a coke inhibitor when impregnated with the zeolite-based support. Therefore, the La-modified zeolite-based catalyst could influence the final product distribution by different selectivity properties. Both zeolites have also different acidity features, which could influence the interaction with La and the HDO reaction mechanism. The low-cost and time-efficient methodology for the catalyst preparation compared to other alternative options would effectively remove the oxygenated compound from the final product and improve renewable diesel production. In addition, the findings in this study would support existing petroleum refineries to produce more sustainable biofuel, hence reducing the initial capital expenditure. Finally, this study would contribute to the body of knowledge and understanding of the behaviour of La-modified zeolite-based catalysts on renewable diesel production.

1.6 Organization of the thesis

This thesis consists of seven main chapters. Chapter 1 is the introduction, which covers the research background, problem statement, research objective, scope of research, the significance of the study, and the organisation of the thesis.

Chapter 2 provides a comprehensive literature review based on previously published research reports on the current trend of the global energy demand, the currently available biofuel sources and the potential feedstocks for renewable diesel production. Additionally, the chapter provides a detailed review of the catalytic hydroprocessing for renewable diesel production, catalyst for hydroprocessing reaction, factors affecting hydroprocessing reaction, deactivation and regeneration of the catalyst and renewable diesel's fuel properties. A summary and research gap are presented at the end of this chapter based on the compiled literature studies.

In Chapter 3, the experimental studies related to objective 1 is provided, which involves the screening process between zeolite beta and HZSM5 as support catalysts based on several process parameter of the HDO reaction of OA for effective diesel-like hydrocarbon production.

Chapter 4 describes the work on objective 2 and 3, specifically on the synthesised La-modified zeolite beta catalyst and its application for renewable diesel production using OA as the model compound for palm products. This chapter also presents the catalyst characterisation using various analytical tools, such as XRD, BET, TPD-NH₃ and TPD-CO₂, and SEM/EDX. Furthermore, the purified liquid product via vacuum distillation was characterised using GC-FID, GC-MS and GC-TCD.

Chapter 5 represents the experimental works based on objectives 2, 3 and 4. In particular, this chapter discussed the HDO reaction of OA over HZSM5 and La-modified HZSM5 for optimal renewable diesel production and the application of optimised reaction conditions to palm-based feedstocks, as well as the reusability of the catalyst. Apart from that, the effect of different La concentrations on the hydrocarbon yield and selectivity of the liquid product is highlighted. The prepared catalysts were characterised using XRD, BET, TPD-NH₃ and TPD-CO₂, SEM/EDX, and TGA. A comparative analysis was performed to evaluate the results from the current study with those of previous findings.

Chapter 6 describes objectives 5, where the catalytic performance of synthesised La₍₁₀₎zeo₍₉₀₎ for the production of diesel-like hydrocarbon fuel from PFAD via HDO. The fuel properties of the generated liquid products were evaluated and compared to that of Ultra-low Sulphur Diesel (ULSD) and petroleum diesel.

Finally, Chapter 7 summarises the significant findings and the main conclusions of the study based on the research objectives. Several recommendations for future work are also provided in this chapter based on limitations of the research findings.



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