UNIVERSITI PUTRA MALAYSIA

NANO-MAGNETIC ACIDIC AND BIFUNCTIONAL CATALYST SUPPORTED ON ACTIVATED CARBON FROM EMPTY FRUIT BUNCH FOR BIODIESEL PRODUCTION

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By

NAEEMAH ABDALABBAS IBRAHIM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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The critical problem arises from the fossil fuels has stimulated recent interests in alternative sources for petroleum-based fuel. An alternative fuel should be technically feasible, readily available, environment acceptable and techno-economically competitive. Biodiesel, which is considered as a potential replacement of conventional diesel fuel is commonly, composed of mono-alkyl ester of long chain that can be prepared from triglycerides which is available in renewable feedstock (vegetable oils or animal fats) utilizing transesterification technology. The feedstock used for the production of biodiesel mainly come from edible vegetable oil which is highly available in most of the countries around the world. However, the competition between food and fuel economies towards the same oil resources may bring global imbalance to the food supply and demand market. The drawbacks of homogeneous catalysts consist of corrosion to the reactor, catalyst separation difficulties, consumption of the catalyst during reaction and production of wastewater. However, conventional catalysts have strong active sites, and is cheap but the difficulty of catalyst separation from reaction is a trending issue that need to be address. To overcome the drawbacks and the limitations of heterogeneous catalysts, magnetic nanoparticle catalysts are often being utilized due to their ease of separation from products, high activity, recyclability and large surface area. This study is responsive to the concern by developing three catalysts based on nonmagnetic oxides and activated carbon (AC) to produce biodiesel from waste, feedstocks. A palm wastes of empty fruit bunches (EFB) was process to acidic fruit empty bunch (AEFB) via hydrolysis and develop into (AC). A hydrothermal reaction at 135 °C to synthesize three catalysts and were tested for biodiesel production using PFAD and WCO. In the first and second catalysts AC-$Fe_{(10)}$- SO_3CI and Na_2SiO_3-NiO-MnO/AC), the conversion of 98 and 96% were obtained while the third catalyst ($CaO_{(10)}$-$Fe_2O_{3(10)}$/AC) the conversion was 95%. All catalysts posed excellent economic viability, since this catalyst is synthesized from waste material, high reusability (6 cycles) and posed with the most effective separation

process of catalyst from biodiesel by magnetization of catalysts (AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC and $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC) due to the high magnetic properties. All the catalysts were characterized to study the morphology (FESEM), functional groups (FTIR), phase and crystallize size (XRD), surface area (BET), vibrating sample magnetometer (VSM), thermal decomposition and stability (TGA) and the number and size of active sites on the surface of the catalysts (TPD-NH_3). According to x-ray diffraction (XRD), the crystal size of AC-$Fe_{(10)}$- SO_3CI, Na_2SiO_3-NiO-MnO/AC and $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC catalysts were found to be 45.21, 39.64 and 32.16 nm, respectively. while the BET surface area was 36.64, 16.80 and 24.96 m^2/g, respectively. TGA shows a loss of decomposition from 772°C -995°C, 289.4°C - 569.2°C and 906°C -991°C, respectively. FESEM images have shown the morphology of the surface, pore sizes, and agglomeration of the catalysts. VSM, determines the magnetization of the catalysts and shows that AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC and $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC has high magnetization of 40.57, 40.27 and 85 emu/g. All three catalysts performed excellently in terms of conversion and demonstrated high magnetic separation from reaction by external magnetic field. The reusability of AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC and $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC catalysts was performed for six cycles before leaching occurred and the catalysts showed high reusability with low metal leaching within the range of the EN 12662 standard specification for contamination content of diesel fuel oil. The produced biodiesel has a kinematic viscosity at 40 ºC of 4.8 and 3.4, flash point of 167 and 134 for PFAD and WCO based biodiesel, respectively which were meets the standard specifications, ASTM D6751 and EN14214 standard. In conclusion, the employment of AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC and $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC magnetic catalysts in esterification/transesterification reaction has significantly enhanced the catalytic activity. The activated carbon generated from agricultural waste (EFB) has demonstrated the ability to support the nonmagnetic catalysts with the display of good magnetic properties. The $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC catalyst showed the best magnetic property while the AC-$Fe_{(10)}$-SO_3CI catalyst showed best conversion. Potentially, these catalysts indicate the ability to be used industrially since there is the efficient catalysts separation from biodiesel section which makes the reduction of extra steps.

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

MANGKIN DWIFUNGSI DAN NANO-MAGNETIK BERASID BERPENYOKONG KARBON TERAKTIF DARIPADA TANDAN BUAH KOSONG KELAPA SAWIT UNTUK PENGHASILAN BIODIESEL

Oleh

NAEEMAH ABDALABBAS IBRAHIM

Februari 2020

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Masalah kritikal yang timbul daripada bahan api fosil telah merangsang minat akhir-akhir ini dalam sumber alternatif bagi bahan api berasaskan petroleum. Bahan api alternatif secara teknikal harus dapat dilaksana, mudah diperoleh, selamat alam sekitar dan kompetit if secara teknoekonomi. Biodiesel, yang dianggap sebagai penggantian bahan api diesel konvensional yang berpotensi biasanya, terdiri daripada rantaian panjang ester monoalkil yang dapat disediakan daripada trigliserida yang boleh diperoleh dalam stoksuapan boleh pulih (minyak sayuran atau lemak haiwan) menggunakan teknologi pentransesteran. Stoksuapan yang digunakan untuk penghasilan biodiesel sebahagian besar datang daripada minyak sayuran boleh makan yang secara meluas terdapat di kebanyakan negara di dunia. Walau bagaimanapun, persaingan antara makanan dan ekonomi bahan api terhadap sumber minyak yang sama mungkin membawa ketidakseimbangan global kepada penawaran makanan dan pasaran permintaan. Kelemahan mangkin yang homogenus terdiri daripada pengakisan reaktor, kesukaran pengasingan mangkin, penggunaan mangkin ketika tindak balas dan penghasilan air sisa. Walau bagaimanapun, mangkin konvensional mempunyai tapak aktif yang kuat, dan murah tetapi kesukaran pengasingan mangkin daripada tindak balas merupakan isu sohor kini yang harus dipertimbangkan. Bagi mengatasi kelemahan tersebut dan limitasi mangkin heterogenus, mangkin nanopartikel magnetik kerap digunakan disebabkan kelancaran pengasingan mereka daripada produk, aktiviti tinggi, kebolehkitaran semula dan kawasan permukaan yang luas. Kajian ini merupakan responsif kepada kepedulian dengan membangunkan tiga mangkin berasaskan oksida bukan magnetik dan karbon teraktif (AC) bagi menghasilkan biodiesel daripada sisa, stok suapan. Sisa tandan buah kosong sawit (EFB) telah diproses menjadi tandan buah kosong berasid (AEFB) melalui hidrolisis dan dihasilkan menjadi (AC). Tindak balas hidrotermal pada 135°C bagi mensintesiskan tiga mangkin dan telah diuji bagi penghasilan biodiesel menggunakan PFAD dan WCO. Bagi mangkin pertama dan kedua, iaitu AC-$Fe_{(10)}$- SO_3CI dan Na_2SiO_3-NiO-MnO/AC),

pengubahan 98 dan 96% telah diperoleh manakala mangkin ketiga (CaO(10)-$Fe_2O_{3(10)}$/AC) pengubahan ialah 95%. Kesemua mangkin memperlihatkan viabiliti ekonomi yang terbaik, disebabkan mangkin tersebut telah disintesiskan daripada bahan sisa, kebolehgunaan semula yang tinggi (6 kitaran) dan digandingkan dengan proses pengasingan mangkin paling efektif daripada biodiesel melalui pemagnetan mangkin (AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC dan $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC) disebabkan sifat magnetik yang tinggi. Kesemua mangkin telah dicirikan bagi mengkaji morfologi (FESEM), kumpulan fungsional (FTIR), fasa dan saiz menghablur (XRD), kawasan permukaan (BET), magnetometer sampel bergetar (VSM), penguraian termal dan stabiliti (TGA) dan bilangan dan saiz tapak aktif ke atas permukaan mangkin (TPD-NH_3). Berdasarkan pembelauan sinar-x (XRD), saiz kristal bagi AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC dan $CaO_{(10)}$-$Fe_2O_{3(10)}$/mangkin AC didapati ialah masing-masing 45.21, 39.64 dan 32.16 nm, manakala kawasan permukaan BET ialah masing-masing 36.64, 16.80 dan 24.96m^2/g. TGA menunjukkan kehilangan penguraian masing-masing daripada 772°C-995°C, 289.4°C-569.2°C dan 906°C-991°C. Imej FESEM telah menunjukkan morfologi permukaan saiz pori, dan pengaglomeratan mangkin. VSM, menentukan pemagnetan mangkin dan menunjukkan bahawa AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC dan $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC mempunyai pemagnetan yang tinggi, iaitu 40.57, 40.27 dan 85emu/g. Semua ketiga-tiga mangkin menyerlah dengan cemerlang dari segi pengubahan dan mengutarakan pengasingan magnetik yang tinggi daripada tindak balas melalui medan magnetik eksternal. Kebolehgunaan semula mangkin AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC dan $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC telah dilaksanakan bagi enam kitaran sebelum pengurasan berlaku dan mangkin menunjukkan kebolehgunaan semula yang tinggi dengan pengurasan logam yang rendah dalam lingkungan julat spesifikasi standard EN 12662 bagi kontaminasi kandungan minyak bahan api diesel. Biodiesel terhasil mempunyai viskositi kinematik pada 40^{o}C dari 4.8 dan 3.4, takat kilat 167 dan 134 masing-masing bagi PFAD dan biodiesel berasaskan WCO, yang memenuhi spesifikasi standard, standard ASTM D6751 dan EN14214. Kesimpulannya, penggunaan AC-$Fe_{(10)}$-SO_3CI, Na_2SiO_3-NiO-MnO/AC dan $CaO_{(10)}$-Fe2O3$_{(10)}$/ mangkin magnetik AC dalam tindak balas esterifikasi/transesterifikasi telah secara signifikan meningkatkan aktiviti pemangkin. Karbon teraktif yang dijana daripada sisa pertanian (EFB) telah memperlihatkan kebolehan untuk menyokong mangkin bukan magnetik dengan penampilan sifat magnetik yang baik. Mangkin $CaO_{(10)}$-$Fe_2O_{3(10)}$/AC menunjukkan sifat magnetik yang baik manakala mangkin AC-$Fe_{(10)}$-SO_3CI menunjukkan pengubahan yang baik. Dari segi potensi, mangkin tersebut memperlihatkan keupayaan untuk digunakan dalam sektor industri disebabkan terdapat pengasingan mangkin yang efisien daripada seksyen biodiesel yang memperlihatkan pengurangan langkah tambahan.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ASTM	American society for testing and materials
XRD	X-ray power diffraction
XPS	X-ray photoelectron spectroscopy
TPD-NH_3	Ammonia-temperature programmed desorption
ACHS-$Fe_{(x)}$	Sulfonated activated carbon supported Iron oxide
TPD-CO_2	Carbon dioxide- temperature programmed desorption
TGA	Thermogravimetric analysis
BET	Brunauer-Emmett-Teller
VSM	Vibrating-sample magnetometer
FT-IR	Fourier-transform infrared spectroscopy
FESEM	Field emission scanning electron microscopy
EDX	Energy-dispersive X-ray spectroscope
EFB	Empty fruit bunches
AEFB	Acidic empty fruit bunch
PKS	Palm kernel shells
OPF	Oil palm fronds
OPL	Oil palm leaf
OPFL	Oil palm frond leaves
OPT	Oil palm trunk
AC	Activated carbon
FFA	Free fatty acid
PFAD	Palm fatty acid distillate
WCO	Waste cooking oil

CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, people are living in an age of great changes. Never before in recorded history had human ever experienced such an enormous rate of development. Consequently, global energy consumption is projected to escalate and is anticipated to reach ca. 30 TW by 2050 (Liu et al., 2010). In other words, "energy is the most pressing issue facing humanity in the coming decades. At present, non-renewable fossil fuels such as coal and oil have been over exploited of energy security. As the reserves of coal and oil have been over-exploited to sustain the vast amount of energy need (Ran et al., 2014). Extensive use of energy derived from limited reserves of fossil fuel will eventually lead to foreseeable depletion, resulting in threatening of energy security as reverses of coal and oil were reckoned to bear out another ca105 and 48 years at end of 2019 (Chen & Wu, 2017). Besides, energy generated from combustion of fossil fuels come at an expense of detrimental greenhouse gases (GHG) emissions which eventually causes critical environmental problems, for instance, global warming and climate changes (Sekizawa et al., 2013). Particularly, GHG emissions associated to energy sector has accounted for 80% of anthropogenic emissions in worldwide scale, which steer a huge influence on the dire environmental crisis (Quadrelli & Peterson, 2007). The increasing global energy demand and environmental related problems thereof have become the driving force in urging the transition of fossil fuels utilization to more promising renewable and clean energy supply. Owing to the global energy consumption continues to escalate and the incapability of energy replenishment from finite sources of fossil fuels to fulfil such needs, a diffusion of energy to more promising renewable carriers is anticipated in the near future. Presently, renewable energy shares an approximate 3.6% of global primary energy mix with a projected rising trend.

Approximate 3.6% of the global primary energy mix with these renewable energies with can be primarily classified as wind (52.14%), solar (17.89%), bio- energy to more renewable energy shares a growth of ca. 13.3% between 2015/2016 interval. Particularly, solar energy displayed the unexploited resource, and exclusion of hydropower power (25.41%), geothermal (4.5%) and ocean energy (0.06%) with an annual capacity highest annual capacity increment of 31.3% which is accounted to 99 GW from 2015 to 2016 (ROESCH, 2018).

The highly abundant solar energy is an unexploited resource and, in this regard, scientists have begun to take advantage of this inexhaustible energy source. Therefore, fossil fuel usage is a major component of the climate change problem. Even though CO_2 capturing and storage (CCS) technology is believed to help alleviate environmental impact of GHG, CCS is inappropriate for mobile application. Therefore, due to this critical problem, government regulations nowdays strongly promote the ulitilization of alternative fuels in the effort to reduce the greenhouse gases (GHG) emission. This problem could be overcome by changing transportation fuel resources from fossil fuel to renewable fuel/biofuel. The benefit not only global climate change, but also local air pollution in metropolitan cities can be prevented by use of renewable fuels (Dahman et al., 2019).

Biomass is a source of electrical or heat energy derived from the plant or animal and can be used as a source raw material in the various industrial processes for a range of products (Li & Liang, 2017). There are four usable types of biomass: 1) wood and agricultural products; 2) solid waste; 3) landfill gas; and 4) alcohol fuels. Most biomass used today is home grown energy.

Malaysia produce about 95.38 million tons of EFB produces solid waste (green weight) annually and could reach 100 million tons of dry weight in 2020 (Umar et al., 2013). All wood and wood residues, oil palm, birch, spruce are the largest source biomass energy (Brosowski et al., 2016). Biomass is used directly as fuel or converted into fuel from plant or animal matters, fibres or industrial chemicals. As biomass such as palm wastes include empty fruit bunches (EFB) and palm kernel shells (PKS) is a natural material that can be recycled; however, it can be also converted into a source of energy (Awalludin et al., 2015). One of the most important uses of biomass is the extraction or production of raw materials and sources containing a high content of carbon (Osman et al., 2016).

In the last years, numerous studies on the potentiality of biomass conversion in various industrial application such as biodiesel production and by the use of biomass-based catalysts. Some of the reported used EFB/$Fe_2(SO_4)_2$ catalyst derived from biomass waste (empty fruit bunches) (Koguleshun et al., 2015); EFB/KOH catalyst (Chuah et al., 2016); PKSB catalyst derived from palm kernel shells (PKS) (Bazargan et al., 2015).

A catalyst is a substance that increases the rate of a reaction without being consumed in the reaction and can continue to act repeatedly. Generally, the chemical reaction can be faster in the presence of a catalyst because the catalyst produces an alternative reaction pathway with lower activation energy in comparison with a non-catalysed mechanism as reaction (Reddy et al., 2013). Catalysts are either alkali, acid, or enzyme catalyst and they might be either homogeneous or heterogeneous (Andreo-Martínez et al., 2018). A homogeneous catalyst composed of molecules dispersed in the same phase (generally gaseous or liquid) as reactant’s molecules. The homogeneous catalysts (acid or alkali) show drawbacks through the need to a large amount of the energy for purifying products and catalyst removal and, as such, they are not reusable.

Catalysts play an essential role in the esterification and transesterification reactions to biodiesel production by speeding up the reaction for higher yield and conversion role of FAME (Narasimharao et al., 2007). As mentioned earlier, the use of the homogeneous catalysts requires a large amount of water because of the washing of biodiesel (Long et al., 2014). On the other side, a heterogeneous catalyst is the one whose molecules are in various phase than reactant's molecules (usually gases or liquids) which can be adsorbed onto the surface of the solid catalyst. It has a large capacity in catalytic both esterification and transesterification reaction from vegetable oils to produce biodiesel than homogeneous catalysts (Serio et al., 2007). The use of heterogeneous catalysts led to simpler, low cost to separation processes, and reduction in water effluent load. Furthermore, it is important to mention that the catalyst not need to be continuously added and can be easier to reuse (Mansir et al., 2017). The main drawback in catalyst process is that the solid-catalytic is still very difficult to the separation from the mixture related to the reuse after filtration through the membrane (Ibrahim et al., 2019). To overcome these challenges, using the magnetic solid catalyst is the best choice because of the magnetic separation generally avoids the loss and increases its reusability in comparison to filtration or centrifugation (Hu et al., 2011). Additionally, as magnetic catalyst properties possess high surface area, large pore volume and high catalytic activity (Erdem et al., 2017). Recently, there is different biomass such as waste material from palm, jatropha, bamboo and others, exploited to produce magnetic catalyst.

The conversion is carried out using different calcinations to carbon because of availability, cheap, and recyclability. An examples are Na_2SiO_3@Ni/C which is active for biodiesel production from bamboo (Zhang et al., 2016); C-SO_3H@Fe/JHC)- the magnetic from Jatropha (Zhang et al., 2017); or 3 aminopropyl trimethoxysilane which is the core-shell structure (Li & Liang, 2017). Briefly, the catalysts are very important compound that were found to be very useful in making the reaction much faster and, meanwhile, they can be used for several times.

The critical problem arises from the fossil fuels has stimulated recent interest in alternative sources for petroleum-based fuel. An alternative fuel should be technically feasible, readily available, environment acceptable and techno-economically competitive. Recently, there has been renewed interest on vegetable oils and animal fats to produce biodiesel. Biodiesel is a liquid fuel similar to petroleum diesel in combustion properties; however, it is essentially sulphur-free, making it a cleaner burning fuel than petroleum diesel. The biodiesel properties are, in many cases, superior to the petro-diesel fuel because the former has higher flash point, ultra-low sulphur concentration, better lubricating efficiency and better cetane number (John et al., 2018). Using biodiesel in a conventional diesel engine substantially reduces air pollutant emissions of hydrocarbons, carbon monoxide, particulate matter, sulfates, polycyclic aromatic hydrocarbons and nitrated polycyclic aromatic hydrocarbons. These reductions are increasing as the amount of biodiesel blended into diesel fuel increases. The best emissions reductions are seen with B100 (100% pure biodiesel without blended with petro-diesel) (Narasimharao et al., 2007).

1.2 Problem statement

A technological strategy that has received special attention is the use and development of heterogeneous catalysts for the production of biodiesel, since these can sometimes be prepared in simple form, present great potential for reuse, and in some cases low cost. In this context, basic catalytic derivatives, acids, bifunctional systems, and enzymatic derivatives, which has shown excellent performance despite the high commercial cost of the purified enzymes, have been evaluated. Despite the advantages of heterogeneous catalysts, there are difficulties, because in some cases these catalysts are very fine powders, which in addition to forming agglomerates during the reaction, part of the catalyst is lost in the separation processes, thus limiting their applications at industrial scale. In this context, the magnetic nanoparticles are particularly attractive as additives to the supports can be used for the heterogeneous catalysts due to their advantages of fast and facile catalyst separation from the reaction mixture by applying an external magnetic field, thereby eliminating process steps such as conventional centrifugation and filtration. However, the magnetic nanoparticles forming particle clusters may restrict the dispersion of the nanoparticles in the reaction mixture due to their magnetic dipole-dipole attraction.

In the biodiesel production, the most important to improve the process efficiency is the catalyst. However, heterogeneous catalyst was used instead of homogeneous catalyst because of the reusable and corrosion problems. On the other hand, the catalytic activity and the stability of the heterogeneous catalyst are the key factors in synthesizing a novel catalyst. To overcome this matter, the carbon-based solid acid magnetic catalysts were introduced in this research, which had proved to have high catalytic activity and good stability.

The typical feedstock used for biodiesel production mainly derived from edible oil that available abundantly around the world. These source materials have led to food vs fuel concerns as conflicts between the needs for biofuel and human food have been reported. The competition between food and fuel economics toward the same oil might bring global imbalance to the food supply and demand on the market. In addition, utilization of edible oil also surely will lead to higher production cost which unattractive for industrial-scale Thus, in order to commercially viable alternative to petroleum derived fuel industry, the use of lower-cost and non-edible oil such as PFAD and WCO are taken into consideration in biodiesel production.

However, the PFAD is an acid oil with large amounts of free fatty acids (FFAs), which the active site of base catalyst for transesterification reaction was normally inhibited by the fatty acid via saponification. Ideally, acid catalysis is a potential candidate for simultaneous esterification of the FFAs and transesterification of the triglycerides to achieve one-pot preparation of FAMEs from PFAD. Therefore, heterogeneous solid acid catalyst provides an environmentally benign and cost-effective process for production of biodiesel from low quality acid oil. However, challenge on developing an efficient solid acid catalyst for one-step esterification-transesterification was still on-going.

1.3 Objectives of the research

The objective of this study is to synthesize active catalyst for esterfaction/transesterification reaction; consist of high surface area, high pore volume, high magnetite and higher stability using activated carbon as the supported material. The synthesized activated carbon based catalysts are then tested in esterification/transesterification reaction of PFAD and waste cooking oil. To achieve this goal, four objectives should be accomplished as follows:

1- Synthesize and characterization of heterogeneous magnetic acid catalyst and bifunctional catalysts.
2- Esterify the PFAD and transesterified the WCO for catalytic activity using the magnetic acid and bifunctional catalysts for biodiesel production.
3- To teste the reusability of synthesized catalysts and characterization of spent catalysts.
4- To determine the fuel properties and characterization of the produced biodiesel.

1.4 Scope of the study

This research focuses on the utilization of palm tree waste to activated carbon (AC) production which is used as nanomagnetic catalyst support. Three different catalysts were developed by doping several metal oxides including Fe, Ni, Mn and Ca on the AC. The preparation of the three catalysts is by the method of wet pore volume impregnation. The three catalysts were individually used in biodiesel production using PFAD and WCO as feedstocks. The methodology of preparing the magnetic catalyst biodiesel was newly developed. This study focuses on esterification using two types of catalysts; and one catalyst for transesterification. The catalysts morphology is studied with FESEM images, the phases and crystal sizes by XRD, the surface area by BET, the acid and base active sites by TPD-NH_3 and TPD-CO_2, the thermal stability by TGA, the magnetic properties by VSM, the phases by XPS and the biodiesel properties by FTIR and ^{1}HNMR. In this work, the screening of the catalysts for optimum biodiesel production are performed with the reusability study and also the characterization of the spent catalysts.

1.5 Thesis outline

This study is separated into five chapters. The first chapter is the introductory chapter which presents the background of the study, problem statement, research objectives, scope of the study, and thesis outline. The second chapter includes the literature review of the several subjects pertinent to the research work. It discussed the literature review of biomass sources of the biomass waste, catalyst synthesis and analysis as explained via previous researchers and study of homogenous and heterogeneous catalysts (solid acid and bifunctional magnetic catalysts), biodiesel production by using heterogeneous basic and acid magnetic catalyst as well as report on using heterogeneous catalyst derived from palm fruit bunch (EFB), parameters affecting catalysts through appear the different support of the catalyst. addition on, utilizing biodiesel feedstock such as (PFAD and WCO) to biodiesel production. The third chapter contains the materials and methods, synthesis and characterization of the solid acid and base magnetic catalyst, reaction method, the study of fuel properties. The fourth chapter shows the experimental results and detailed discussions and analysis of the results. Lastly, the fifth chapter concluded the thesis and recommendation for the future research study.

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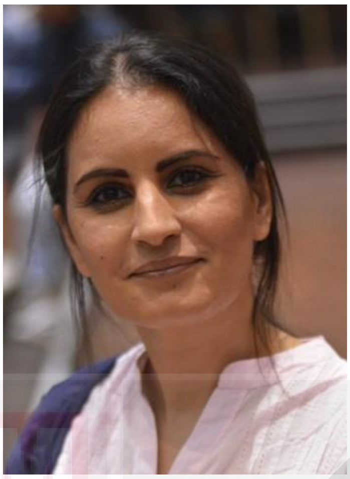

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