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PRODUCTION OF GREEN DIESEL FROM SATURATED CHICKEN FAT OIL CATALYZED BY BINARY METAL OXIDE SUPPORTED ON MULTI-WALLED CARBON NANOTUBES

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By

NURUL ALIANA BINTI NASHARUDDIN

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

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Fossil fuels have been the most important energy and fuel sources over centuries. However, there has been growing distressed regarding on energy crisis caused by the oil reserve depletion and the effect of environmental issues (e.g. acid rain and global warming). Due to the high demand for energy, renewable energy has gained extensive attention worldwide in the past ten years as alternative energy to replace fossil fuels. In particular, fuels obtained from biomass (biofuels) has become a great option as a sustainable substitute for fossil fuels. Deoxygenation processes that exploit milder reaction conditions under H_2-free atmospheres appear environmentally and economically effective for the production of green diesel. However, the presence of a catalyst in the deoxygenation reaction is important to excite optimum catalytic activity of the synthesized catalyst for a specific reaction system. The catalyst support plays an essential role in synthesizing catalyst, which is to improve the interaction between active metal-support, promoting active metal dispersion on the surface and providing the adequate active site. Herein, green diesel was produced by catalytic deoxygenation of chicken fat oil (CFO) over oxides of binary metal pairs (Ni–Mg, Ni–Mn, Ni–Cu, Ni–Ce) supported on multi-walled carbon nanotubes (MWCNTs). The yield of hydrocarbons are arranged in the order of blank < MWCNT < Ni_{10}/MWCNT < Ni_{10}-Mn_{10}/MWCNT < Ni_{10}-Cu_{10}/MWCNT < Ni_{10}-Mg_{10}/MWCNT < Ni_{10}-Ce_{10}/MWCNT. The deoxygenation reaction will lead to the formation of C15 and C17 of diesel fractions as the main product. Thus, the *n*-(C_{15}+C_{17}) selectivity are arranged in the increasing order of blank < MWCNT ~ Ni_{10}-Cu_{10}/MWCNT < Ni_{10}-Ce_{10}/MWCNT < Ni_{10}-Mn_{10}/MWCNT < Ni_{10}-Mg_{10}/MWCNT < Ni_{10}/MWCNT. The result shows that Ni_{10}/MWCNT has highest *n*-(C_{15}+C_{17}) selectivity but with low hydrocarbon yield due to its favor toward cracking-decarboxylation/decarbonylation (deCOx) reaction. Therefore, presence of Mg and Mn with Ni seems effective in deoxygenation activity, with hydrocarbon yields of >75% and *n*-(C_{15}+C_{17}) selectivity of >81%, indicating that deCOx of CFO is favored by the existence of the high amount of lower strength strong acidic sites along with noticeable

strongly basic sites. Based on a series of studies of different Mg and Mn dosages (5–20 wt %), the oxygen free-rich diesel-range hydrocarbons produced efficiently by Ni_{10}-Mg_{15}/MWCNT and Ni_{10}-Mn_5/MWCNT catalysts yielded >84% of hydrocarbons, with *n*-(C_{15}+C_{17}) selectivity of >85%. The findings reveal that Ni_{10}-Mg_{15}/MWCNT shows high resistancy toward coke formation (coke < 5 wt %) under TGA analysis. In addition, Ni_{10}-Mg_{15}/MWCNT shows high catalytic stability and reusability up to 5 cycles with >73% of yield and *n*-(C_{15}+C_{17}) selectivity of >66%.

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

PENGHASILAN DIESEL HIJAU DARIPADA MINYAK LEMAK AYAM TEPU DI PANGKIN OLEH PASANGAN LOGAM OKSIDA PADA NANOTIUB KARBON BERBILANG-DINDING

Oleh

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Bahan api fosil telah menjadi tenaga dan sumber bahan api yang paling penting sejak berabad-abad. Walau bagaimanapun, terdapat kerisauan yang semakin meningkat mengenai krisis tenaga yang disebabkan oleh kekurangan rizab minyak dan kesan daripada isu-isu alam sekitar (contohnya hujan asid dan pemanasan global). Oleh kerana permintaan yang tinggi, tenaga yang boleh diperbaharui telah mendapat perhatian luas di seluruh dunia sejak sepuluh tahun yang lalu sebagai tenaga alternatif untuk menggantikan bahan api fosil. Khususnya, bahan api diperoleh daripada biojisim (bahan api bio) telah menjadi pilihan yang baik sebagai pengganti bahan api fosil yang mapan. Proses penyahoksigenan yang mengeksploitasi keadaan tindak balas yang sederhana di bawah atmosfera bebas H_2 muncul secara mesra alam sekitar dan berkesan dari segi ekonomi untuk pengeluaran diesel hijau. Walau bagaimanapun, kehadiran pemangkin dalam tindak balas penyahoksigenan adalah penting untuk merangsang aktiviti pemangkin secara optimum oleh pemangkin yang disintesis bagi sistem tindak balas tertentu. Sokongan pemangkin memainkan peranan penting dalam mensintesis pemangkin, yang bertujuan untuk meningkatkan interaksi antara logam aktif-penyokong, mempromosikan penyebaran logam aktif di atas permukaan dan menyediakan tapak aktif yang mencukupi. Di sini, diesel hijau telah dihasilkan oleh penyahoksigenan pemangkin bagi minyak lemak ayam (CFO) menggunakan pasangan logam oksida (Ni–Mg, Ni–Mn, Ni–Cu, Ni–Ce) yang disokong pada nanotiub karbon berbilang-dinding (MWCNTs). Hasil hidrokarbon disusun mengikut urutan tanpa pemangkin < MWCNT < Ni_{10}/MWCNT < Ni_{10}-Mn_{10}/MWCNT < Ni_{10}-Cu_{10}/MWCNT < Ni_{10}-Mg_{10}/MWCNT < Ni_{10}-Ce_{10}/MWCNT. Tindak balas penyahoksigenan akan membawa kepada pembentukan C15 dan C17 daripada pecahan diesel sebagai hasil utama. Oleh itu, pemilihan *n*-(C_{15}+C_{17}) disusun mengikut urutan meningkat tanpa pemangkin < MWCNT

~ Ni_{10}-Cu_{10}/MWCNT < Ni_{10}-Ce_{10}/MWCNT < Ni_{10}-Mn_{10}/MWCNT < Ni_{10}-Mg_{10}/MWCNT < Ni_{10}/MWCNT. Hasil menunjukkan Ni_{10}/MWCNT mempunyai pemilihan *n*-(C_{15}+C_{17}) yang tertinggi tetapi dengan hasil hidrokarbon yang rendah kerana ia lebih memihak kepada tindak balas pemecahan-penyahkarbosilan/penyahkarbonilan (deCOx). Oleh itu, kehadiran Mg dan Mn dengan Ni kelihatan efektif dalam aktiviti penyahoksigenan, dengan hasil hidrokarbon >75% dan pemilihan *n*-(C_{15}+C_{17}) >81%, menunujukkan bahawa deCOx bagi CFO digemari disebabkan kehadiran jumlah yang tinggi bagi kekuatan rendah tapak asid kuat bersekali dengan tapak bes kuat yang sangat ketara. Berdasarkan siri kajian pelbagai dos Mg and Mn (5–20 wt %), hidrokarbon oksigen bebas tinggi dalam lingkungan diesel terhasil dengan cekap oleh pemangkin Ni_{10}-Mg_{15}/MWCNT dan Ni_{10}-Mn_5/MWCNT menghasilkan >84% hidrokarbon, dengan pemilihan *n*-(C_{15}+C_{17}) >85%. Penemuan ini menunjukkan bahawa Ni_{10}-Mg_{15}/MWCNT mempunyai daya ketahanan yang tinggi terhadap pembentukkan kok (kok <5% berat) dibawah analisis TGA. Di samping itu, Ni_{10}-Mg_{15}/MWCNT menunjukkan kestabilan pemangkinan yang tinggi dan boleh diguna semula sehingga 5 kitaran dengan >73% hasil dan pemilihan *n*-(C_{15}+C_{17}) sebanyak >66%.

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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 Master. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ASTM	American society for testing and materials
EDX	Energy dispersive X-Ray spectroscope
DO	Deoxygenation
HDO	Hydrodeoxygenation
deCOx	Decarboxylation/decarbonylation
FFA	Free fatty acid
FT-IR	Fourier transform infrared
GC	Gas chromatograph
GC-MS	Gas chromatograph mass spectroscopy
TPD-NH_3	Ammonia-temperature programmed desorption
TPD-CO_2	Carbon dioxide-temperature programmed desorption
FESEM	Field emission scanning electron microscopy
TGA	Thermogravimetric analysis
XRD	X-ray diffraction analysis
MWCNTs	Multiwalled-carbon nanotubes
CFO	Chicken fat oil
TGs	Triglycerides
BE	Binding energy

CHAPTER 1

INTRODUCTION

1.1 Overview on Biomass, Biofuel and Green Diesel Production

In a global society, the production of fuels from renewable feedstock has gained great attention as an alternative energy source to substitute fossil fuels (coal, petroleum, and natural gas). This is due to the extensive utilization of fossil fuels that leads to the depletion in fossil fuels reserves and consequently, giving rise to the environmental impact, such as global warming, acid rain, and greenhouse gas emission (Rani, 2014) which contributed to the environmental health hazard. It further escalates with the growing economy and the increasing population that lead to the overwhelming demand in energy. Hence, to fulfil the demand and overcome the deficiency of energy, the renewable sources have been used to replace the non-renewable energy resources as an alternative measure (Satyarthi, 2011).

Biomass is one of the renewable sources which is abundant naturally, and typically being produced as a waste from the agriculture sector, such as sugarcane bagasse, corn, paddy husks, woody biomass, wheat, walnut shell, and bark (Lim *et al*., 2012; Pradhan *et al*., 2019; Saidur *et al*., 2011; Shafie *et al*., 2012). Recently, biomass-derived triglycerides-based feedstock has been utilized as an alternative to renewable raw material. Triglycerides are mainly made up of long chains fatty acid esters, which make up the structure of vegetable oils and fats. The triglycerides are composed of three fatty acids either in the form of saturated, monounsaturated, and polyunsaturated (Sotelo-Boyas *et al*., 2012). Typically, triglycerides are derived from vegetable oil, waste cooking oil and animal fats. Hence, biomass-derived triglycerides is a suitable feedstock for conversion into biofuel as an alternative clean hydrocarbon fuel which is sustainable and reliable to replace conventional fuels (Dale, 2008; Demirbas, 2007). The benefits of biofuel are due to its renewable properties, largely available from various biomass sources, biodegradable and environmental-friendly a low-emission of CO_2 and NO_x upon combustion of fuel. There is various type of biofuels such as biodiesel, bio-hydrogen, bio-gas, bio-methane, bio-ethanol, green gasoline, and green diesel. Among these biofuels, green diesel offered great interest from the researchers.

Green diesel is also known as the second generation of biofuel, which made up of hydrocarbon chain that has a similar structure with petroleum-derived fuels. Even though biodiesel or fatty acid methyl esters (FAMEs) has been established widely as the most well-known biofuel, but biodiesel has a different chemical structure from petroleum-derived fuels due to the presence of oxygenated compound (Bezergianni *et al*., 2010). The high-oxygenated compound exists in biodiesel resulting in undesirable properties such as high viscosity, low oxidative stability, a high cloud point, nitrogen oxide (NO_x) emissions, and low energy-density (Orozco *et al*., 2017). In contrast, green diesel that is comprised of mainly hydrocarbon and free oxygenated species has better properties such

as higher heating value, lower density, lower viscosity and greater oxidative stability (Neonufa *et al*., 2017; Santikunaporn & Malee, 2012). Due to these reasons, green diesel exhibits the most promising biofuel in replacing conventional fuels due to its outstanding fuel properties than biodiesel.

Deoxygenation processes are particularly attractive for producing green diesel, and the operational costs are relatively lower than the current upgrading process used in existing petroleum refineries, the hydrodeoxygenation (HDO) process. The hydrodeoxygenation process involves direct conversion of fatty acids via removal of oxygenated species, retaining the number of carbon atoms, with H_2O as a by-product under H_2 atmosphere (Hermida *et al*., 2015). Meanwhile, deoxygenation processes involve the removal of oxygenated species in the forms of CO, CO_2 and H_2O via decarbonylation and decarboxylation (deCOx) under H_2-free atmosphere (Asikin-Mijan *et al*., 2017; Echaroj *et al*., 2015). The hydrocarbon products formed typically contain one less carbon atom (C_{n-1}) than the original fatty acid chain. The green diesel produced from deoxygenation exhibits better fuel characteristics with high heating value (HV), high cetane number (80–90), lower viscosity and high fuel stability. Thus, it has been widely accepted by many research studies that green diesel is the most promising substitute for fossil-based diesel (Douvartzides *et al*., 2019).

1.2 Chicken Fat Oil as Bio-based Feedstock

Selecting the appropriate feedstocks for green diesel production is extremely important for industrial practices. Typically, feedstock has been chosen based on the criteria of (1) wide-availability of the feedstock, (2) economical manageability and (3) geographical for easy access of feedstock. Usually, vegetable oil feedstock used for biofuel production consists of edible oil and non-edible oil. However, edible oils facing problems related to the competition between food and fuel issues. Meanwhile, non-edible oil such as jatropha oil, rubber oil, ceiba oil, karanja oil, sterculia oil and castor oil are expensive which make it not suitable to be used in the production of biofuel (Khan *et al*., 2014). Thus, the production of green diesel derived from animal fats has become a great interest. Animal fats that are commonly used are beef tallows, chicken fat, mutton fat and pork lard (Boey *et al*., 2011; Hoque *et al*., 2011; Jeong *et al*., 2009; Mutreja *et al*., 2011). Among these, chicken fat oil (CFO) offers a better alternative renewable source**.** Commonly, chicken which is known as staple food worldwide has a production of about 107.1 million metric tonnes (Seidavi *et al*., 2019). It was estimated that fat contents in chicken poultry are approximately 10-12% (Alptekin *et al*., 2014). Thus, there will be around 10.7-12.9 million metric tons of chicken fat produced per year**.** The CFO comprised of C_{16} (palmitic acid) and C_{18} (oleic acid) fatty acids. It was estimated that deoxygenation of CFO via decarboxylation/decarbonylation (deCOx) reaction as the main pathway will lead to the formation of the hydrocarbon, mostly made up of diesel fractions (*n*-C_{15} and *n*-C_{17}) (Kaewmeesri *et al*., 2015).

1.3 Deoxygenation Catalyst

Metal oxides seem to be realistic deoxygenation catalysts for the future. Among those metal oxide catalysts, Ni metal as the main catalyst shows activity comparable to noble metals in converting lipid-based feeds to liquid hydrocarbons. As discussed by Morgan *et al*., Ni on carbon support catalysts showed similar activity to that of Pd- and Pt-promoted catalysts at higher concentration, which suggested that Ni has good performance in replacing noble metals in deoxygenation (Morgan *et al*., 2010). Although Ni seems promising in deoxygenation, it is favored toward excessive cracking reaction that resulting in lower yields of diesel range hydrocarbon products and catalyst deactivation. In an attempt to enhance the deoxygenation activity of the Ni catalyst, oxides of other metals, such as Ce, W, Co, Fe, Cu, Mo, Zn, Mg and Ca, have also been used as a promoter in deoxygenation under H_2-free conditions (Aysu *et al*., 2016; Loe *et al*., 2016; Rezgui & Guemini, 2005; Tani *et al*., 2011). These promoters were added into the main catalyst to form the binary metal oxide catalysts. Interestingly, Ce and Cu offer better deoxygenation reaction selectivity in diesel-like hydrocarbon production. Aysu *et al*. (2016) studied the deoxygenation of jojoba oil over Ce-promoted catalysts and the results showed that the reaction occurred exclusively via deCOx, which yielded higher percentages of aliphatic compound. A similar case, with Cu-promoted catalysts studied by Loe *et al*., whereby the Cu-promoted catalyst was demonstrated to be active in removing the oxygen atoms from free-fatty-acid-derived oxygenates and yielded >90% of diesel-range hydrocarbons (Loe *et al*., 2016). Additionally, a basic promotion catalyst (MgO) also favored the deCOx reaction. Tani *et al*. (2011) discovered that MgO-supported catalysts resulted in enhanced triglyceride cracking via decarboxylation and the green diesel produced resembled conventional liquid fuel. Moreover, the use of basic metal catalysts can suppress coke formation and offer greater catalyst stability (Asikin-Mijan *et al*., 2015).

Instead of catalyst promoter, the catalyst support always plays a critical role in promoting the deoxygenation reaction. This is due to the support being able to enhance active metal dispersion, simultaneously increasing the active sites for catalysis of the reaction (Murali Dhar *et al*., 2003). Carbon is promising support, which can be attributed to the high-specific area and the nature of carbon itself, being thermally stable; thereby minimizing the sintering of the active metal during the deoxygenation reaction (Zhao *et al*., 2013). Nano-sized carbon supports, such as multi-walled carbon nanotubes (MWCNTs) have specific pore structures that offer better thermal stability (Zhang., 2009) than micron-sized activated carbon supports; and the use of MWCNTs as a catalyst support in the deoxygenation reaction has been recognized. As discussed by Asikin-Mijan *et al*., MWCNTs were used as catalyst support in the deoxygenation of *Jatropha curcas* oil, resulting in high selectivity toward C_{15}+C_{17} via the deCOx pathway and producing >80% hydrocarbon yield (Asikin-Mijan *et al*., 2018). Based on the finding it can be suggested that the use of MWCNT as catalyst support offers better catalytic stability, thus, increased the catalytic activity.

1.4 Problem Statement

Even though hydrodeoxygenation (HDO) pathway typically preferred by the refinery industry but hydrodeoxygenation process involves direct conversion of fatty acids via removal of oxygenated species, retaining the number of carbon atoms, with H_2O as a by-product under high pressure of H_2 atmosphere. Due to the absorbent cost of H_2 consumption during the reaction process, makes the hydrodeoxygenation process becomes less preferable to be used for the production of green diesel. Hence, production of green diesel via deoxygenation has been proposed since the reaction proceed under H_2-free atmosphere. Deoxygenation process is typically driven by the presence of a catalyst. There is various type of catalyst that has been used, including noble metals, sulfided catalyst and metal oxides in promoting deoxygenation. Noble metals such as Pd, Pt, and Rh were proven to be the most active metal catalysts of deoxygenation activity but yet high-cost constraints made them unattractive. Besides, the sulfided catalyst also has the disadvantage to be used as a catalyst owing to sulfur leaching. Hence, research has been focused on generating catalyst that is inexpensive and essentially free of sulfur. Metal oxides seem to be realistic deoxygenation catalysts for the future. Therefore, NiO catalyst has been developed as the main catalyst to perform the deoxygenation reaction. This is due to the NiO shows high reactivity for upgrading the triglycerides-based biomass into diesel-range alkanes as main products. Besides that, catalyst support plays an important role in improving the metal dispersion and facilitating deoxygenation reaction effectively. Carbon-based support catalyst has been proven to be effective in enhancing the deoxygenation of fatty-acid and its derivatives. MWCNT as one of the carbon-based support with nano-sized has gained great interest in recent times due to its high surface area to volume ratio affordability. Even though NiO supported MWCNT is effective for deoxygenation, but the catalyst still highly favourable toward coke formation leading to decrease the catalytic activity (Abdulkareem-Alsultan *et al*., 2019). Nevertheless, the addition of metal oxide promoter such as Mg, Mn, Cu and Ce in NiO supported on MWCNTs to form binary metal oxide catalyst will assist in enhancing the catalytic deoxygenation process. The promoter with the basic property known as coke inhibitor and has most promotional effect in retarding the catalyst deactivation. Thus, the present study highlights on the development of a series of oxides binary metal pairs (Ni–Mg, Ni–Mn, Ni–Cu, Ni–Ce) supported on MWCNTs in the deoxygenation of chicken fat oil (CFO) under H_2-free conditions.

1.5 Objectives

The objectives of conducting this research are

1. To synthesize a series of oxides binary metal pairs (Ni–Mg, Ni–Mn, Ni–Cu, Ni–Ce) supported on MWCNTs and to characterize the physicochemical properties of catalysts.
2. To produce green diesel from chicken fat oil via catalytic deoxygenation pathways under the reaction condition at 350°C for 2 h reaction time using 3 wt% catalyst loading under inert flow.
3. To study the catalyst stability and reusability Ni_{10}-Mg_{15}/MWCNT.

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