



UNIVERSITI PUTRA MALAYSIA

***DEVELOPMENT OF SUPPORTED NICKEL-BASED CATALYSTS FOR
DEOXYGENATION OF WASTE COOKING OIL TO RENEWABLE FUEL
PRODUCTION***

WAN NOR ADIRA BINTI WAN KHALIT

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By

WAN NOR ADIRA BINTI WAN KHALIT

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

March 2022

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

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March 2022

Chair : Professor Datuk ChM. Ts. Taufiq Yap Yun Hin, PhD
Faculty : Science

The development of renewable diesel fuel from the deoxygenation of non-edible oil is an alternative to non-renewable fuels. This study investigated the catalytic deoxygenation of waste cooking oil (WCO) over supported Ni-based catalysts. The deoxygenation of WCO was conducted using different types of supports: activated carbon (AC), reduced graphene oxide (rGO), and beta zeolite (Zeo). The addition of Ni to AC improves the physicochemical properties of the catalyst, owing to the high number of acid-base sites, high surface area, smaller crystallite size, and high pore volume of the catalyst. Based on the catalytic results, Ni₂₀/AC was the most active catalyst, which achieved 90% hydrocarbon yield and 89% selectivity towards *n*-(C₁₅+C₁₇). Furthermore, it was stable up to the fourth cycle with consistent hydrocarbon yield (85-87%) and 66-77% selectively towards *n*-(C₁₅+C₁₇). Further investigation was conducted to study the effect of bifunctional catalysts (NiLa, NiCe, NiFe, NiMn, NiZn, and NiW) supported on AC. High hydrocarbon yield above 60% with lower oxygenated species was found in the liquid product with the product selectively toward *n*-(C₁₅+C₁₇)-diesel fractions. The predominance of *n*-(C₁₅+C₁₇) hydrocarbons with concurrent production of CO and CO₂ indicated that the deoxygenation pathway preceded via decarbonylation and decarboxylation mechanisms. For NiLa/AC, high deoxygenation activity with better *n*-(C₁₅+C₁₇) selectivity was obtained due to great synergistic interaction between La-Ni, and its compatibility of acid-base sites increased the removal of oxygenates. For the effect of La on the deoxygenation performance, it was found that a high percentage of La species would be beneficial in the removal of C-O bonded species. Furthermore, optimum deoxygenation activity of 88% hydrocarbon yield with 75% *n*-(C₁₅+C₁₇) selectivity was obtained over 20% La, which strongly evinced that La leads to more significant enhancement of deoxygenation activity. The NiLa/AC reusability study showed consistent deoxygenation with 80% hydrocarbon yield and 60% *n*-(C₁₅+C₁₇) hydrocarbons selectivity within six runs. As the NiZn/AC catalyst also showed high performance in deoxygenation activity, the optimization over a series of Ni₂₀Zn_x/AC catalysts (X: 5–20 wt.%) was also studied. The Ni₂₀Zn₁₀/AC catalyst exhibited superior deoxygenation activity by yielding 86% hydrocarbons and 79% of *n*-(C₁₅ + C₁₇)

selectivity. High deoxygenation activity is corroborated by the higher acidity and basicity strength of the catalyst and the oxygenate species removal that occurred via decarbonylation pathway. The Ni₂₀Zn₁₀/AC catalyst showed promising catalytic stability and reusability up to four runs with hydrocarbon yield (78 – 87%) and *n*-(C₁₅ + C₁₇) selectivity within the range of 43 – 70%, respectively. The decrease in the *n*-(C₁₅ + C₁₇) selectivity in the fourth cycle was due to the active metal species leaching and coking. In conclusion, all Ni-based catalysts demonstrated significant catalytic activity and reusability for green diesel production.



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

**PEMBANGUNAN MANGKIN BERASASKAN NIKEL YANG DISOKONG
UNTUK PENYAHOKSIGENAN SISA MINYAK MASAK KEPADA
PENGHASILAN BAHAN API YANG BOLEH DIPERBAHARUI**

Oleh

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Pembangunan bahan bakar diesel yang boleh diperbaharui melalui penyahoksigenan minyak yang tidak boleh di makan merupakan satu alternatif kepada bahan bakar yang tidak boleh diperbaharui. Kajian ini menyiasat penyahoksigenan pemangkin bagi sisa minyak masak (WCO) menggunakan pemangkin berasaskan Ni. Tindak balas penyahoksigenan terhadap WCO dilakukan menggunakan pelbagai jenis sokongan: karbon teraktif (AC), grafena oksida yang dikurangkan (rGO) dan beta zeolit (Zeo). Penambahan Ni ke AC meningkatkan sifat fizikokimia pemangkin yang baik disebabkan oleh bilangan tapak asid-bes yang tinggi, luas permukaan yang tinggi, saiz kristal yang lebih kecil dan jumlah liang pemangkin yang tinggi. Dari hasil penyahoksigenan, Ni₂₀/AC menunjukkan pemangkin paling aktif dengan memberikan 90% hasil hidrokarbon dan 89% pemilihan *n*-(C₁₅+C₁₇). Tambahan pula, pemangkin tersebut menunjukkan kestabilan sehingga kitaran keempat dengan hasil hidrokarbon yang konsisten (85-87%) dan pemilihan *n*-(C₁₅+C₁₇) (66-77%). Penyelidikan lebih lanjut dilakukan untuk mengkaji kesan pemangkin dwifungsi (NiLa, NiCe, NiFe, MiMn, NiZn, dan NiW) yang disokong pada AC. Hasil hidrokarbon melebihi 60% dengan spesies oksigen yang lebih rendah didapati dalam produk cecair dengan pemilihan utama terhadap *n*-(C₁₅+C₁₇)-pecahan diesel. Penguasaan hidrokarbon *n*-(C₁₅+C₁₇) dengan pengeluaran CO dan CO₂ secara serentak menunjukkan bahawa penyahoksigenan melalui tindak balas penyahkarbonilan dan penyahkarbosilan. Penyahoksigenan aktiviti yang tinggi dan pemilihan *n*-(C₁₅+C₁₇) yang lebih baik oleh pemangkin NiLa/AC disebabkan oleh interaksi sinergistik yang baik antara La-Ni, dan keserasian tapak-tapak asid dan bes meningkatkan lagi penyingkiran spesies oksigen. Kajian mengenai kesan La terhadap penyahoksigenan aktiviti dijalankan dan mendapati bahawa peratusan spesies La yang tinggi bermanfaat untuk penyingkiran spesies terikat C-O. Di samping itu, 20% La menunjukkan optimum penyahoksigenan aktiviti dengan menghasilkan 88% hasil hidrokarbon and 75% pemilihan *n*-(C₁₅+C₁₇), yang membuktikan bahawa La membawa kepada peningkatan penyahoksigenan aktiviti yang lebih ketara. Kajian kebolegunaan semula NiLa/AC menunjukkan tindak balas penyahoksigenan yang stabil dengan hasil hidrokarbon 80% dan pemilihan *n*-(C₁₅+C₁₇) 60% dalam enam kitaran. Oleh kerana

pemangkin NiZn/AC juga menunjukkan penyahoksigenan aktiviti yang tinggi maka pengoptimuman terhadap satu siri pemangkin Ni₂₀Zn_x / AC (X: 5-20 wt.%) juga dijalankan. Pemangkin Ni₂₀Zn₁₀/AC menunjukkan penyahoksigenan aktiviti yang tinggi dengan menghasilkan 86% hidrokarbon dan 79% pemilihan *n*-(C₁₅+C₁₇). Aktiviti penyahoksigenan yang tinggi disebabkan oleh kekuatan asid dan bes pemangkin dan penyingkiran spesis oksigen berlaku melalui tindak balas penyahkarbonilan. Diperhatikan, pemangkin Ni₂₀Zn₁₀/AC menunjukkan penyahoksigenan aktiviti yang stabil dan penggunaan semula pemangkin sehingga empat kitaran dengan hasil hidrokarbon (78-87%) dan pemilihan *n*-(C₁₅+C₁₇) (43-70%). Penurunan pemilihan *n*-(C₁₅+C₁₇) adalah disebabkan oleh pelarutlesapan tapak aktif logam dan pembentukan karbon. Kesimpulannya, semua pemangkin berasaskan Ni menunjukkan aktiviti pemangkinan dan penggunaan semula yang ketara untuk pengeluaran diesel hijau.



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LIST OF ABBREVIATIONS

OVAT	One variable at a time
ASTM	American Society for Testing and Materials
AOCS	American Oil Chemists' Society
JCPDS	Joint Committee on Powder Diffraction Standard
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
TPD-NH ₃	Temperature Programmed Desorption - ammonia
TPD-CO ₂	Temperature Programmed Desorption - carbon dioxide
TCD	Thermal Conductivity Detector
FESEM	Fields Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray
GC-FID	Gas Chromatography-Flame Ionization Detector
GC-MS	Gas Chromatography-Mass Spectrometry
FTIR	Fourier Transform Infrared Spectrometry
CHNOS	Carbon, Hydrogen, Nitrogen, Oxygen, and Sulphur
IUPAC	International Union of Pure and Applied Chemistry

CHAPTER 1

INTRODUCTION

1.1 Research Background

Global population growth causes rapid industrialisation, expanding urbanisation, and an increase in the number of transportations, resulting in increased global energy consumption. According to the US Energy Information Administration, global energy consumption is expected to increase by 50% between 2010 and 2050, (Sourmehi, 2021). Currently, fossil fuels meet the majority of energy demand, with oils, coal, and natural gas accounting for 33%, 27%, and 24%, respectively (Gross, 2020; Looney, 2020). However, these are not considered renewable energy sources because their applications are unfavourable from a financial, environmental, and ecological standpoint.

The transportation sector is essential to globalisation and contributes significantly to the economy. The primary sources of transportation energy use worldwide are gasoline and diesel. Global transportation fuel demand is relatively high, with approximately 4.9 billion L of gasoline and diesel consumed daily (Kalghatgi, 2019). Unfortunately, this consumes most fossil fuels, negatively affecting the living environment. The world's fossil fuel reserves are also rapidly depleting (Pattanaik & Misra, 2017). One of the most severe problems caused by fossil fuels is the massive emission of greenhouse gases (GHGs) that contribute to global warming.

According to the Inventory of U.S. Greenhouse Gas Emissions and Sinks (2021), carbon dioxide (CO₂) accounted for 80% of GHG emissions in 2019, followed by methane (10%), nitrous oxide (7%), and fluorinated gases (3%) (Figure 1.1a). Transportation was the leading source of GHG emissions (29%), followed by electricity (25%), industry (23%), commercial and residential (13%), and agriculture (10%) (Figure 1.1b). Researchers worldwide are concerned about the rapid increase in CO₂ emissions from transportation. One of the primary reasons for over 1.2 billion passenger cars worldwide is the proliferation of automobiles (Kalghatgi, 2018). According to the ITF Transport Outlook, (2019), global transportation demand is expected to triple by 2050.

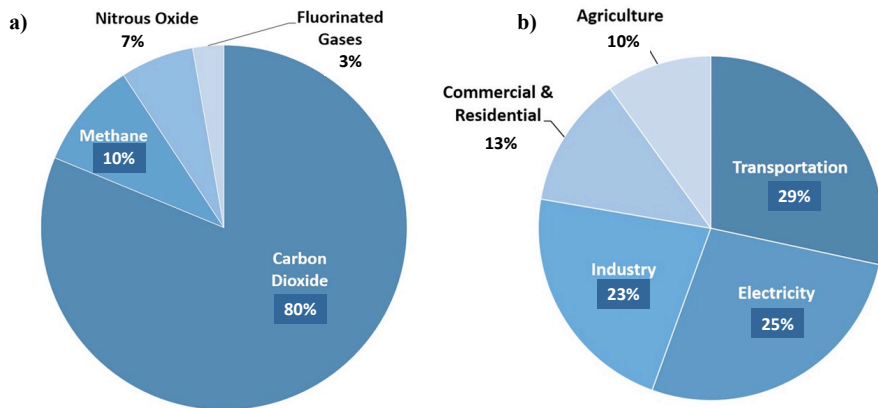


Figure 1.1: Overview of (a) GHGs emission and (b) sources of GHGs in 2019
 [Adapted from Inventory of U.S. Greenhouse Gas Emissions and Sinks (2021)]

A substantial amount is consumed from various fossil fuel resources to meet this energy demand. Unfortunately, current consumption rates will deplete crude oil and natural gas reserves in 33 and 41 years, respectively. Every year, we consume approximately 11 billion tonnes of oil. However, crude oil reserves are depleted at a rate of 4 billion tonnes per year. If current trends continue, oil reserves will be depleted by 2051.

Nonetheless, if increased gas production can fill the energy gap left by oil, those reserves will last an additional eight years until 2061. Even though the world has enough coal reserves to last until the end of the century, increased production is needed to fill the void left by the depletion of oil and gas reserves. Coal reserves will supply enough energy until 2088. Even so, the global energy consumption rate is not constant because it grows significantly with the global population and improved living standards. Furthermore, the world is on the verge of an energy crisis, prompting researchers to investigate alternative approaches. Figure 1.2 depicts the world's coal, oil and gas energy reserves and the point at which fossil fuels may be depleted in the future.

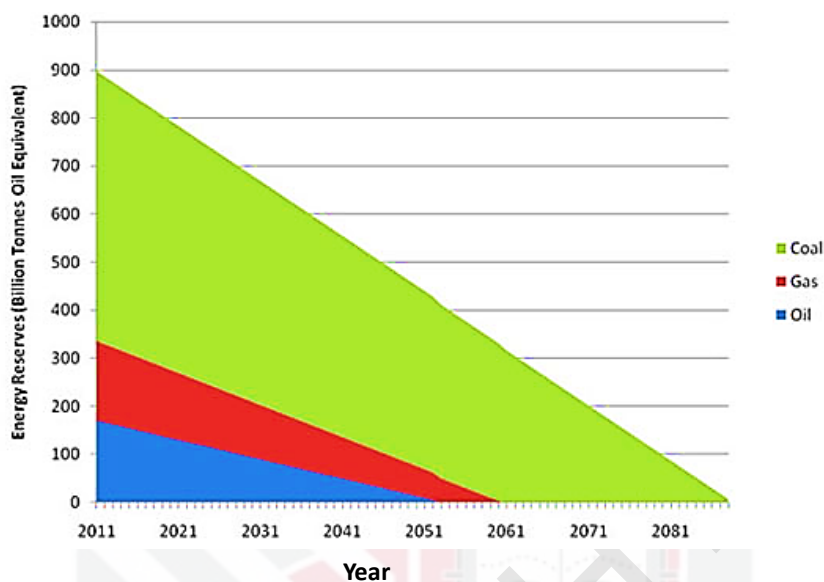


Figure 1.2: Energy reserves for coal, gas, and oil in the future (Kuo, 2019)

1.2 Renewable Energy

Using renewable energy resources to replace fossil fuels would reduce GHG emissions and make renewable energy more appealing. Renewable energy is considered “green” and is one of the environmentally friendly energies that never run out and can be used without reducing its future availability. Hydropower, solar energy, geothermal, wind energy, marine energy, and biomass energy are the primary sources of renewable energy (Pattanaik & Misra, 2017). Currently, the primary focus of renewable energy research is on biomass energy. Agriculture crop residue, forestry, food processing, and animal fats are all biomass sources. Waste vegetable oils and animal fats have been targeted as a renewable feedstock for biofuel production (Kordulis et al., 2016).

Figure 1.3 depicts global energy consumption by energy sources in 2020. Renewable energy accounts for 12% of global energy consumption. Biomass energy (39%) is one of the most important renewable energy sources. Renewable energy, such as biofuels, can improve energy security while reducing GHG emissions and preventing air pollution. Future generations will benefit from a cleaner environment, a more stable economy, and a more reliable energy source as more renewable resources are used. According to Baležentis et al. (2019), a 1% increase in biomass energy output results in a 0.089% reduction in GHG emissions.

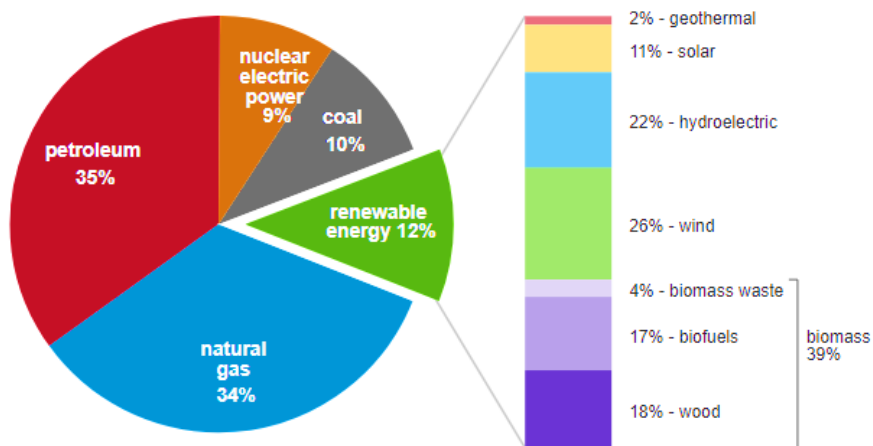


Figure 1.3: Global energy consumption by energy sources in 2020 [Adapted from U. S. Energy Information Administration (2021)]

1.3 Biofuels as a Petroleum Alternative

Biofuels are liquid or gaseous fuels derived from biomass such as corn starch, vegetable oils, animal fats, sugarcane, grasses, or trees. Biofuels are the only viable alternatives to petroleum because they have the potential for large-scale production due to the abundance of biomass sources. Furthermore biofuels are biodegradable and contribute to environmental sustainability. Pyrolysis, gasification, and chemical and biochemical processes are all methods for producing biofuel from biomass. Syngas (CO and H_2) is produced by pyrolysis and gasification and is converted to hydrocarbons for use as fuels. Chemical processing of biomass yields biofuels via homogeneous or heterogeneous catalysis, whereas biological processes (for example, biomass fermentation) use enzymatic catalysis.

Green fuel is a biofuel that includes green gasoline ($\text{C}_8\text{-C}_{14}$) and green diesel ($\text{C}_{15}\text{-C}_{18}$) that contain *n*-alkane and *n*-alkene hydrocarbons. The green fuel obtained is similar to gasoline and diesel from crude oil refining in petroleum refineries. Green diesel production is a simple, cost-effective, and low polluting process. Deoxygenation is the most effective method for producing green diesel from biomass when heterogeneous catalysts are used. Furthermore, green diesel, which has a higher heating value, energy density, cetane number, and quality than diesel fuel, was recently named the most preferred biofuel.

1.4 Problem Statement

Excessive waste cooking oil (WCO) production has become a major global issue. Improper WCO handling may stimulate the production of organic contaminants in land and water, contributing to environmental pollution. The proper recycling of WCO through conversion to industrial products such as green diesel is an ideal solution. WCO can be used directly in a diesel engine. However, there are several disadvantages to using WCO directly including high viscosity, low volatility, and engine problems such as coking on injectors, carbon deposition, and lubricating oil thickening (Biswas et al., 2017). As a result, WCO must be upgraded to be used as green diesel. Due to its similar diesel-fuel properties, high cetane number, and high calorific value producing green diesel using hydroprocessing technologies is a potential process for producing diesel-like hydrocarbon (Gamal et al., 2020).

Hydroprocessing technology (e.g., hydrodeoxygenation) is preferred in the refinery industry. Adding H₂ results in forming hydrocarbons in the C₁₅ - C₁₈ range and removing O₂ as a by-product in the form of H₂O. However, due to the high cost of H₂ consumption, hydrodeoxygenation is less desirable method of producing green diesel. As a result, the production of green diesel via deoxygenation has been implemented, with the reaction taking place in an H₂-free environment.

However, current deoxygenation research is more focused on using noble metals (such as Pt and Pd) and sulfided metals (such as ReNiMoS, NiMoS, CoMoS, and NiWS) as catalysts (Lup et al., 2017a). These active metals have been proven to be the most effective catalysts. Nonetheless, due to the high cost of noble metals, large-scale green diesel production is limited, and sulphur leaching from sulfided catalysts has the potential to contaminate green diesel quality (Ramesh et al., 2020). Hence, research has focused on developing low-cost and sulfur-free catalysts. The transition Ni approximately 1750 and 3450 times less expensive than the noble metals Pd and Pt (Asikin-Mijan et al., 2018; Santillan-Jimenez et al., 2013).

In addition, Ni-based catalysts also outperformed numerous noble-metal catalysts (Hongloi et al., 2021). Thus, transition metal oxides (TMO) are more suitable for deoxygenation. Ni-based catalysts have been studied to improve deoxygenation activity among various TMO catalysts. This is due to the high reactivity of Ni-based catalysts in converting triglycerides-based biomass to primary products in the diesel-range hydrocarbon. On the other hand, it has been proposed that the primary role of Ni-based catalysts is to promote C-C and C-O cleavage via decarboxylation/decarbonylation pathways. Evidently, a 60Ni/Al₂O₃ catalyst was effectively upgraded the triglycerides in sunflower oil to green diesel with 96% conversion to hydrocarbon and 52% to *n*-C₁₇ selectivity (Gousi et al., 2017).

Nevertheless, in the deoxygenation of soybean oil, a 20 wt.% Ni/C catalyst also showed a greater conversion to hydrocarbon (92%) followed by 5 wt.% Pd/C (30%) and 1 wt.% Pt/C (23%) (Morgan et al., 2010). Previously, the deoxygenation of triolein over Ni/HMS catalyst was capable of inducing the C-C cleavage and C-O cleavage and resulted in a

higher selectivity towards diesel-range (C_{11} - C_{20}) (95%) (Zulkepli et al., 2018). The acidic sites of the Ni-based catalysts is also responsible for effectively removing oxygenating species in deoxygenation reaction.

Furthermore, larger pore diameters of catalysts may make waste oil molecules more easily transported to active catalytic sites, thus increasing deoxygenation activity. The nature of the reactant, on the other hand, has minimal effect on the activity of Ni-based catalysts. As a result, Ni-based catalysts are considered suitable for a wide range of reactants in the production of green diesel, particularly in WCO.

Catalyst supports are also crucial in increasing deoxygenation activity. Carbon-based support effectively promotes the deoxygenation of fatty acid and their derivatives. Activated carbon (AC) also has a large surface area due to its microporous structure, which contains numerous functional oxygen groups on the surface, thereby improving its efficacy as an oxygen removal agent (Zhao et al., 2017). Furthermore, it aids in the reduction of undesirable reactions such as polymerization, cracking, and coke formation. Although the deoxygenation activity of the Ni-supported AC catalyst is prominent, the catalyst still produces highly undesirable coke formation.

Thus, incorporating metal oxides promoters such as Mn, W, La, Zn, Fe, and Ce to into a binary system could reduce the deactivation reaction rate. It has been proposed that the synergistic interaction of the acidic and basic sites derived from these metals species may allow for improve catalytic stability and deoxygenation activity. Therefore, the current research focuses on developing supported Ni-based heterogenous catalysts on AC for the catalytic deoxygenation of WCO in an H_2 -free environment.

1.5 Objectives of the Study

The primary goals of this research are as follows:

1. To synthesise and characterise the physicochemical properties of Ni-based catalysts using the wet impregnated method with other transition metals and various supports.
2. To examine the catalytic performance of the synthesised catalysts and optimise the deoxygenation reaction under various catalytic parameters.
3. To assess the reusability and stability of the catalysts for catalytic deoxygenation of WCO.
4. To determine the fuel properties of green diesel based on the American Society for Testing and Materials (ASTM) method.

1.6 Scope of the Study

The wet-impregnation method was used in this study to synthesise Ni-based heterogenous catalysts. First, the deoxygenation reaction of Ni-based catalysts was investigated using various supports (AC, reduced graphene oxide (rGO), and beta zeolite (Zeo)). The physical properties of the catalysts were determined using XRD, BET, TPD-NH₃, TPD-CO₂, TGA, FESEM-EDX, and XPS. The performance of deoxygenation was evaluated to select the best catalyst with high catalytic activity. The liquid product was tested using GC-FID, GC-MS, GC-TCD, FTIR, and CHNOS. Further research was conducted to investigate the impact of acid-base bimetallic catalysts. Various metals, including Ce, Mn, La, Fe, Zn, and Fe were doped with Ni and screened for the incredible bimetallic catalysts, which produced high diesel-ranged hydrocarbon.

The potent catalysts were used in optimisation studies with a one-variable-at-a-time approach to study the effect of different catalyst loadings, reaction temperatures, and reaction times. The catalysts were then tested for reusability and stability studies. Catalyst reusability studies are critical for estimating the economics of catalysts for large-scale production. Under favourable reaction conditions, the reusability activity was evaluated. Renewable fuel properties are also discussed.

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