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DEVELOPMENT OF SUPPORTED CARBON-BASED CATALYST FOR PRODUCTION OF GREEN DIESEL VIA DEOXYGENATION OF FREE FATTY ACIDS

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By

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

The introduction of green diesel produced from the deoxygenation of non-edible feedstock is an alternative to conventional fuels. Hereby, the evaluation of catalytic deoxygenation of palm fatty acid distillate (PFAD) was carried out in an environment free of H_2 to produce green diesel over mono-metallic (Co & Mn) and bimetallic catalysts (Co-Mo, Co-Ag, and Mo-Ag) supported on activated carbon (AC) derived from waste coconut shells. The biomass-derived AC prepared from waste coconut shells offers a competitive edge from the aspect of production cost. Based on the catalytic deoxygenation activity, the $Co_{(10wt.)}$/AC catalyst showed a higher yield and selectivity than the $Mn_{(10wt.)}$/AC. It was found that $Co_{(10wt.)}$/AC catalysts exhibited high deoxygenation activity with hydrocarbon yield (C_8-C_{20}) was (71%) and (C_{15}+C_{17}) selectivity was (46%), attributed to strong acid–base-sites, which consecutively favouring C–O bond cleavage through the deoxygenation route. Further studies were carried out on bimetallic catalysts (Co-Mo, Co-Ag, and Mo-Ag) on AC supports. The effect of Mo in bimetallic catalyst $Co_{10}Mo_n$/AC at various concentrations (n=5–20 wt.%) was investigated on the deoxygenation reactions performance. Based on the study results, the bimetallic catalyst $Co_{(10wt.)}$-$Mo_{(10wt.)}$/AC exhibited high catalytic performance with 92% hydrocarbon components (C_8-C_{20}) yield and 89% selectivity for (C_{15} +C_{17}). This is owing to the good physicochemical properties of the catalyst, such as high strong acid-base sites, high crystallite size, good surface area and pore volume. Furthermore, it was stable until the sixth run maintaining hydrocarbon diesel components yield and selectivity of (C_{15} +C_{17}) >80%. On the other side, the Co-Ag/AC and Mo-Ag/AC catalyst performed well in deoxygenation reactions, the optimization of a series of $Co_{(10wt.\%)}$-$Ag_{(Z)}$/AC and $Mo_{(10wt.\%)}$-$Ag_{(n)}$/AC catalysts (z & n: 5–20 wt.%) was also investigated. Astoundingly, the bimetallic catalyst $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC and $Mo_{(10wt.\%)}$-$Ag_{(20wt.\%)}$/AC exhibited a synergistic effect between the active metals Co-Ag and Mo-Ag with the activated carbon support (AC). The aforementioned catalysts have amazing physicochemical properties such as high surface area, high porosity, good dispersion of active metals on the support, strong acid and base density. These properties significantly

facilitated the selective deoxygenation (deCOx) pathway of the fatty acids by exhibiting the greatest hydrocarbon (C_8–C_{20}) fractions yield of 92% & 93% and selectivity of (C_{15}+C_{17}) 95% & 90%. In addition, the $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC and $Mo_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC catalysts also exhibit high stability and can be reused for up to eight cycles by producing hydrocarbons (C_8 - C_{20}) ~ 75-90 % and selectivity (C_{15}+C_{17}) ~ 70-90 %. Moreover, these catalysts showed an excellent coke inhibition with less than 5 wt.% of coke determined by TGA analysis. Thus, it can be believed a potentially promising catalyst for the production of green diesel, at the same time providing economic opportunities and added value to the palm oil industry. In summary, the bimetallic catalysts $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC and $Mo_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC showed high catalytic activity represented in superior yield and selectivity besides distinguished reusability.

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

PEMBANGUNAN MANGKIN BERASASKAN PENYOKONG KARBON UNTUK PENGHASILAN DIESEL HIJAU MELALUI PENYAHOKSIGENAN ASID LEMAK BEBAS

Oleh

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Pengerusi : Profesor Datuk ChM. Ts. Taufiq Yap Yun Hin, PhD
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Pengenalan diesel hijau yang dihasilkan daripada penyahoksigenan bahan mentah yang tidak boleh dimakan adalah alternatif kepada bahan api konvensional. Dengan ini, penilaian penyahoksigenan pemangkin bagi distilat asid lemak sawit (PFAD), minyak jarak (JCO), dan sisa minyak masak (WCO) telah dijalankan dalam persekitaran bebas H_2 untuk menghasilkan diesel hijau dengan menggunakan mangkin logam (Co & Mn) dan pasangan logam (Co-Mo, Co-Ag, dan Mo-Ag) yang disokong pada karbon aktif (AC) yang diperoleh**i** daripada sisa tempurung kelapa. AC dari bio-jisim yang disintesis daripada sisa tempurung kelapa menawarkan kelebihan daya saing dari aspek kos pengeluaran. Berdasarkan aktiviti penyahoksigenan, mangkin $Co_{(10wt.)}$/AC menunjukkan hasil dan pemilihan yang lebih tinggi daripada $Mn_{(10wt.)}$/AC. Didapati bahawa mangkin $Co_{(10wt.)}$/AC mempamerkan aktiviti penyahoksigenan yang tinggi dengan 71% penghasilan hidrokarbon (C_8-C_{20}) adalah dan 46% pemilihan (C_{15}+C_{17}) yang disebabkan oleh bilangan tapak asid-bes yang tinggi dan memihak kepada pemecahan ikatan C–O melalui laluan penyahoksigenan. Kajian susulan telah dijalankan ke atas mangkin pasangan logam (Co-Mo, Co-Ag, dan Mo-Ag) pada penyokong AC. Kesan Mo dalam pasangan mangkin logam $Co_{10}Mo_n$/AC pada pelbagai kepekatan (n=5–20 wt.%) telah dijalankan melalui tindak balas penyahoksigenan. Berdasarkan keputusan kajian, mangkin pasangan logam $Co_{(10wt.)}$-$Mo_{(10wt.)}$/AC menunjukkan prestasi yang tinggi dengan menghasilkan 92% komponen hidrokarbon (C_8-C_{20}) dan 89% pemilihan (C_{15} +C_{17}). Ini disebabkan oleh sifat fizikokimia mangkin seperti bilangan tapak asid-bes kuat yang tinggi, saiz kristal yang tinggi, luas permukaan dan liang mangkin yang baik. Tambahan pula, mangkin ini menunjukkan kestabilan yang tinggi dan boleh digunakan semula sehingga enam kitaran dengan mengekalkan hasil diesel hidrokarbon dan pemilihan (C_{15} +C_{17}) >80%. Selain itu, mangkin Co-Ag/AC dan Mo-Ag/AC menunjukkan prestasi yang baik dalam tindakbalas penyahoksigenan, oleh itu, pengoptimum terhadap satu siri mangkin $Co_{(10wt.\%)}$-Ag($_Z$)/AC dan Mo($_{10wt.\%}$)-Ag ($_n$)/AC (z & n: 5–20 wt.%) telah dijalankan. Hasil kajian menunjukkan mangkin pasangan logam $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC dan $Mo_{(10wt.\%)}$-$Ag_{(20wt.\%)}$/AC mempamerkan interaksi

sinergistik antara logam aktif Co-Ag dan Mo-Ag dengan penysokong karbon aktif. Mangkin tersebut mempunyai sifat fizikokimia yang mengagumkan seperti luas permukaan yang tinggi, keliangan yang tinggi, penyebaran logam aktif yang baik pada sokongan, asid kuat dan ketumpatan bes. Sifat-sifat ini memudahkan laluan deCOx terpilih bagi asid lemak dengan menghasilkan pecahan hidrokarbon (C_8–C_{20}) sebanyak 92% & 93% dan pemilihan (C_{15}+C_{17}) sebanyak 95% & 90%. Selain itu, pemangkin $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC dan $Mo_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC juga mempamerkan kestabilan yang tinggi dan boleh diguna semula sehingga lapan kitaran dengan penghasilan ~75−90% hidrokarbon (C_8–C_{20}) dan pemilihan (C_{15}+C_{17}) sebanyak 70–90 %. Di samping itu, mangkin ini menunjukkan pembentukan kok yang sangat baik dengan kurang daripada 5% berat kok yang ditentukan oleh analisis TGA. Justeru itu, mangkin ini berpotensi digunakan untuk menghasilkan diesel hijau dan pada masa yang sama menyediakan peluang ekonomi dan nilai tambah kepada industri minyak sawit. Secara ringkasnya, pemangkin pasangan logam $Co_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC dan $Mo_{(10wt.\%)}$-$Ag_{(10wt.\%)}$/AC menunjukkan aktiviti mangkin yang tinggi dalam penghasilan hidrokarbon dan pemilihan (C_{15}+C_{17}) selain kebolehgunaan kitaran semula mangkin.

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This thesis was submitted to the Senate of the 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

AC	Activated carbon
ASTM	American society for testing materials
BET	Brunauer-Emmett-Teller
BJH	Barrett–Joyner–Halenda
BTL	Biomass to liquid
CNSL	Cashew nut shell liquid
COD	Chemical oxygen demand
CPO	Crude palm Oil
CHNOS Analysis	Analysis for determination of carbon, hydrogen, nitrogen, sulfur and oxygen
CPO	crude palm oil
CO2-TPD	temperature-programmed desorption of CO_2;
DCO	Decarbonylation
DCO2	Decarboxylation
deCOx	Deoxygenation
DPO	Degummed Palm Oil
DTG	Differential Thermogravimetry
FAME	Fatty Acid Methyl Ester
FESEM-EDX	Field Emission Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy
FTIR	Fourier Transform Infrared
FFA	Free Fatty Acids
FAME	FAME, fatty acid methyl ester;
FFA	FFA Free Fatty Acids

FWHM	Full width at half maximum
GDP	Gross domestic product
GHG	Green House Gas
GC-FID	Gas Chromatography–Flame Ionisation Detection
GC-MS	Gas Chromatography–Mass Spectrometry
HDO	Hydrodeoxygenation
JCO	Jatropha Carcass Oil
OPEC	Organization of the Petroleum Exporting Countries
OVAT	One-variable-at-a time
PFAD	PFAD Palm Fatty Acid Distillate
TCD	Thermal conductivity detector
TGA	Thermo Gravimetric Analysis
TMO	Transition metal oxides
TG	TAG, triacylglycerol;
WCO	Waste cooking oil
XPS	X-ray photoelectron spectroscopy
XRD	X-ray Powder Diffraction
XRF	X-ray fluorescence

CHAPTER 1

INTRODUCTION

1.1 An Overview to Sustainable Energy, Biofuel and Green Diesel

Global energy consumption expanded at a pace of 2.9% in 2018, nearly twice the 10-year average of 1.5%, and the fastest since 2010, but slowed down again to 1.3% in 2019. Energy consumption growth rates, represented by natural gas and renewable energy (Looney, 2020; Spencer, 2019), have increased despite modest GDP (Gross domestic product) growth and rising energy prices. The use of fossil resources as a main feedstock to produce fuels and chemicals along with the growing environmental issues concerns about greenhouse gas emission and the depletion of petroleum reserves, spurred the society to utilize alternative resources.

The current rapid development in the road transport sector is driving the demand for diesel oils. The global request for diesel oil is growing swifter than any other petroleum product where diesel oil has been projected to experience an increase of about 33.2 million barrels daily in the year 2040, as compared to the 27.5 million barrels per day that was recorded in 2015 (Nilsson, 2016) . As regarding transportation related activities, biofuels are known to be the only substitutes that support neutrality of carbon and are thus becoming paramount progressively, due to their ability to easily integrate into the existing infrastructure (Lup et al., 2017). Furthermore, to mitigate the dependability on fossil fuels, liquid transport fuel production obtained from renewable sources, is expanding globally. There have been many efforts to produce biofuels, however, most of them focused on single-component fuels such as ethanol, butanol, and recently, there has been an increase in research tailored at producing long-chain hydrocarbons that can be consumed in diesel engines (Lee and Nikraz, 2015; Snunkhaem Echaroj, 2015).

More so, researches on biofuels have already commenced incorporation to the transport sector, with the inclusion of bioethanol as well as biodiesel, which comprise fatty acid methyl esters (FAMEs); as this depends on in-edible vegetable oil, lignocellulosic, and waste materials. Biodiesel is an inexhaustible fuel substitute that has the ability of being consumed in a diesel motor, either being blended with petroleum-diesel fuel or in a pure state. With regards to ecological appraisal and sustainability, biodiesel assists in the reduction of particulate emission, as well as in cutting down greenhouse gases emission so as to lessen air defilement. However, biodiesel (FAME) comprises higher oxygen content that makes it experience less density in energy as well as reduced oxidation stability, as compared to petroleum diesel (Lup et al., 2017).

Since the last decade, different technologies have been evolved to produce renewable diesel from biomass materials to serve as a replacement for petroleum diesel. Thus, the deoxygenation of biomass to form hydrocarbons has obtained considerable attention as a promising technology that can help in producing green diesel due to the resemblance of the attained hydrocarbons properties with that of the petroleum diesel. Additionally, the costs of operational processes are less than the present process utilized in present-day petroleum refineries, thus the need for the process of hydrodeoxygenation (HDO) has emerged. Moreover, the process of hydrodeoxygenation involves the direct formation of bio-oil through eliminating of oxygen atoms, resulting in the preserving the carbon atom number in the parent compounds, with water as a by-product in the H_2 ambient (Ameen et al., 2017). From an economic point of view, deoxygenation is beneficial because it cuts the costs related to utilization of H_2 in the hydrodeoxygenation process. In general, the deoxygenation of fatty acids is performed through two major reaction pathways to produce green diesel, i.e., decarbonylation and decarboxylation (deCOx), whereby (i) the decarbonylation (DCO) pathway involves the elimination of carbonyl groups through C-C and C-O bond scission to produce straight-chain alkenes by releasing of CO and water as by-products (Oi et al., 2020) (Eq. 1), and (ii) the decarboxylation (DCO_2) pathway, which involves the elimination of carboxylic groups through C-C bond scission and the release of CO_2 to produce a straight-chain alkane with one carbon atom less than the original fatty acid (Eq. 2)

Decarbonylation pathway

$$R\text{-}COOH \rightarrow R'\text{-}CH=CH_2 + CO_{(g)} + H_2O \qquad \text{(Eq. 1.1)}$$

Decarboxylation pathway

$$R\text{-}COOH \rightarrow R\text{-}H + CO_{2\,(g)} \qquad \text{(Eq. 1.2)}$$

R, R′ = alkyl groups

Consequently, deoxygenation is one of the pioneer treatment procedures utilized to upgrade bio-oil into green diesel. Thus, the bio-oil upgrading via deoxygenation process renders as a practical choice desired to produce oxygen free green diesel with better properties like higher heating value, lower density, lower viscosity, and greater oxidation stability (Hongloi et al., 2022). Due to these reasons, green diesel exhibits the most promising biofuel replacement for conventional fuels and with outstanding fuel properties better than biodiesel. Furthermore, green diesel originating from biological sources resembles petrol-diesel-like fuels and adheres to ASTM D975 specifications, which is not mono-alkyl esters (Munoz et al., 2012).

In light of the above proposition, the community is forced to develop a new technology for obtaining biofuels by benefitting from the availability of renewable raw materials (Datta and Mandal, 2016). The suitable feedstock selection for production of green diesel is exceptionally vital for industrial implementations. Generally, feedstock selection has been subjected to the following criteria (1) availability of the feedstock, (2) economic feasibility, and (3) geographically easy to access feedstock. Commonly, edible and inedible vegetable oils are used for biofuel production. However, edible oils encounter some difficulties associated with fuel-food competitions. Various natural vegetable oils and animal fats have proved to be promising renewable resources. Amongst them, fatty acids are the fundamental constituents of plant and animal oils/fats such as waste cooking oil (Romero et al., 2016), refined palm oil (Srifa et al., 2014), rapeseed oil (Lovás et al., 2015) and palm fatty acid distillates (PFADs) (Kiatkittipong et al., 2013). Since Malaysia is considered among the largest producers of crude palm oil (CPO), refining a large amount of crude palm oil will generate a massive amount of PFAD – 780,000 t ("Essential Palm Oil Statistics Palm Oil Analytics," 2017).

Meanwhile, oils that are inedible such as jatropha oil, Karanja oil, rapeseed oil and rubber oil are expensive for use in the production of biofuel (Ooi *et al.*, 2019; Asikin-Mijan. *et al.*, 2020). Biofuels derived from renewable sources like vegetable oils and biomass can be used a as substitute fuel, lowering the amount of fossil fuels consumed and thus greenhouse gas emissions (Isa and and Ganda, 2018; Ooi et al., 2019). Liquid biofuels made from renewable bio-resources like free fatty acids (FFAs) feedstock have attracted great interest in the recent times as is evident from the literature published in this field. (Asikin-Mijan et al., 2020; Wu et al., 2017). Amid inedible vastly utilized vegetable oils used in producing biofuels, comprises the PFAD – one that is increasingly used especially in South East Asia. Furthermore, in the crude palm oil (CPO) refining process, free fatty acids are excluded as by-products and are called palm fatty acid distillates (PFAD). The PFAD is not of human-edible grade, it is utilized as feedstock in soaps and the oleochemical industry, and as fuel for local power/process heat. PFAD is a promising feedstock for green diesel production and it is cheaper than edible oils, it has a high fatty acid content >85wt.% (Ibrahim et al., 2019). PFAD contains large amounts of saturated palmitic acid and mixed C_{18} acids. This is in addition to the abundance of supply, flexibility, and low price, which makes PFAD a promising and desirable renewable feedstock for green diesel production that will reduce emissions of greenhouse gases (Baharudin et al., 2020).

Regarding green diesel production via deoxygenation reactions, the catalyst is considered as an important factor in acquiring the main products with high yield and selectivity. The ability of catalysts could be enhanced via optimization of the operational conditions, treatment, as well as modification procedures. The treatment activity contributes to the creation of sites with stronger, hyper-active Brønsted-Lewis acid-base as well as magnified crystal sizes that can help enhance deoxygenation efficacy, reusability, as well as selectivity in producing green diesels of high quality, yet lesser oxygen components (Mahdi et al., 2021). The choice of active metal catalyst for deoxygenation processes is critical since it has a crucial impact on product yield and quality. Though many researches have investigated fatty acids deoxygenation alongside plant oils, all were limited to the use of special catalysts. In a study by Snare et al., a group of metals were investigated (Pd, Pt, Ru, Ni, Rh, Ir, and Os), and these were purportedly supported either on carbon or metal oxides (Pattanaik and Misra, 2017). Eventually, they concluded that fatty acids, as well as their esters, are transformed directly into straight-chained hydrocarbons via the process of deoxygenation are favourable particularly on activated carbon-supported Pd and Pt metals. Nevertheless, the high expense of the aforementioned metals is unfavourable compared to catalysts that are more convenient for deoxygenation from the economic perspective (Pang et al., 2019). Consequently, the restriction with regards to noble metals being made available across the globe has spurred the need to investigate other inexpensive catalysts such as transition metals Ni, Co, Cu, Mo, Ag, Zn, Mn and W for biomass upgrading. (Cheah et al., 2021; Ramesh et al., 2019). Among the transition metals, Co exhibited excellent decarboxylation activity by the formation of saturated unbranched compounds, in addition to unsaturated olefinic compounds via decarbonylation pathways, which led to the production of water as a by-product (Asikin-Mijan et al., 2018). Srifa *et al.*, (2014) studied palm oil deoxygenation for the production of green diesel over monometallic catalysts (Co, Ni, Pd, and Pt), which is also supported on γ-Al_2O_3. Notably, the results revealed that the Co-based catalyst tends toward the decarbonylation route as well as/or either the decarboxylation which has similarity to the reaction of hydrodeoxygenation. Meanwhile, Ni, Pd, and Pt-based catalysts favoured the reactivity of the decarbonylation process far beyond that of hydrodeoxygenation. However, Pd and Pt noble metals show great HDO activity, yet are generally selective towards aromatic ring hydrogenation rather than deoxygenation. Consequently, Ni catalyst was favoured towards excessive cracking reaction, which lowers the diesel range hydrocarbon yields and causes catalyst's deactivation due to its high acidity. Surprisingly, the Co-based catalyst seems promising in deoxygenation, as it showed high catalytic performance in the deoxygenation reaction through its excellent acidic–basic sites. Júlia de Barros Dias Moreira et al. (2020) studied the Macauba acid oil deoxygenation over a Co-base catalyst supported on activated carbon. From the results, it was discovered that there was an occurrence reactivity via deCOx, yielding around 96% bio-hydrocarbons, in the range of both green diesel as well as kerosene (Moreira et al., 2020). To reinforce the catalytic activity of a Co-based catalyst, oxides of selected metals like Mo, W, Fe, Ca, Mn, Ag, and Ni have been used as a promoter in deoxygenation under H_2-free conditions (Choo et al., 2020). The aforementioned promoters were added to the main metal to form the binary metal oxide catalysts.

Agricultural residues are beneficial in providing raw materials for the preparation of catalytic supports, and the use of cheap and highly effective metals opened the way to apply them in deoxygenating different kinds of animal fats as well as vegetable oils (Mahdi et al., 2021). As a substitute of catalyst promoter, a major role is usually played by the catalyst support in enhancing the reactivity of the deoxygenation process. This is due to the ability of the support to develop dispersion of metals, while synonymously increasing the active sites on the catalyst (Aliana-Nasharuddin et al., 2019). Furthermore, carbon is deemed an auspicious support, one that could be attributable to specific area, alongside the carbon structure itself, in a thermally stabilized order; thence, reducing the active metal sintering in the course of deoxygenating reactivity (Wang et al., 2018). Activated carbon (AC) as catalyst support preserves a brilliant future because of its distinguished physical properties such as lower coke formation propensit, high surface area, as well as thermal stability, which helps in mitigating active metal sintering in the course of deoxygenation reaction.

In literature, only a few investigations on the catalytic deoxygenation of rich-fatty acids feedstock on carbon-based bimetallic catalysts are present. Kiatkittipong *et al.*, (2013) in their study, informed that higher catalytic activities were exhibited by the Pd/C catalyst in the hydrotreatment of fatty acid distillates (PFAD), where the diesel-range products yield was >80%, while it showed weak catalytic activity with crude palms triglyceride, Degummed Palm Oil (DPO), as well as Crude Palm Oil (CPO), respectively. In reference to the findings, it could be conclusively stated that AC as a catalyst support, provides better catalytic stability, and thus increased deoxygenation activity.

1.2 Problem Statements

The growth of motorization and industrialization worldwide, has led to high petroleum-based fuel demand (Liu et al., 2015). Today, the wellsprings of petroleum fuel are drained and depleted, which had caused the increment in fossil fuel price and makes the supplanting of fossil fuel with biofuel more critical than ever. However, biofuel conveys a significant expense tag in the industry because of the costly feedstock and high maintenance. Therefore, the use of inedible waste contributes to reducing the cost of biofuel production. Furthermore, there has been a recent development among technologies like the vegetable oil hydrotreatment as well as the biomass to liquid fuels (BTL) in producing green diesel, coming up with promising routes to fulfil future energy soaring demands (Arun et al., 2015). Catalytic hydrotreating reaction simultaneously expels sulphur, nitrogen, and oxygen as hydrogen cleaves bonds from carbon-heteroatom or carbon-carbon, in a molecule. Meanwhile, the hydrodeoxygenation (HDO) process is typically applied in the refinery industry which involves direct conversion of fatty acids, where molecules of organic–oxygen that are found in feedstocks, undergo hydrogen-based reactions at high temperatures (250–400 °C) as well as pressure (3–10 MPa). The obtained products are hydrocarbon paraffin-based-biofuels free of oxygen, preserving the carbon atoms number with a byproduct of H_2O (Arun et al., 2015). Therefore, the hydrodeoxygenation process is less preferable in green diesel production due to cost of consumed H_2 during the reaction phase. Otherwise, fatty acids deoxygenation, is proceeded by reactivity phases of both decarbonylation (DCO) and decarboxylation (DCO_2) respectively to yield green diesel products by removal of oxygen as CO_2/CO via a direct cleavage of the C–C bond (Krobkrong et al., 2018). Despite the DCO route final product having one carbon less compared to the feedstock, it is more preferred than the HDO. The process is carried out with the exception of an external source of H_2, of which there is self-generated H_2 gas via water-gas-shift in the course of the reaction, thus facilitating a reaction of hydrogenolysis (Xing et al., 2018), alongside the inert atmospheric condition mitigating the cost of using a high-pressure equipment.

Regarding green diesel production, the catalyst is a critical factor in improving the proficiency of the process. Heterogeneous catalysts, which proved a high capability and functionality were used. Besides the catalytic activity and stability, the heterogeneous catalysts are the key factors in synthesizing a novel catalyst. Of late, many researchers have studied catalytic deoxygenation for several fatty acids feedstock, that can help in producing liquid hydrocarbons using different catalysts such as noble metal catalysts, which comprise of a high catalytic activity in the deoxygenation reaction with or without hydrogen gas, but economic limitations due to high production costs make them unappealing (Hongloi et al., 2022). Meanwhile, the sulphide metal catalyst has some drawbacks which are accountable for the contamination of the final product with sulphur coupled with the issue of corrosion, because sulphidation (oxides to sulphides) occurred in the active sites of the catalyst surface (Khan et al., 2019). To outperform these difficulties, transition metal oxides have proved to be of special significance regarding their catalytic performance and unique properties, such as sustainability, minimal quantity required, reusability, and presence of both basic and acidic properties (Cheng et al., 2016). Amongst those, the monometallic type has caught phenomenal attention in the catalytic deoxygenation of fatty acids, specifically Fe (Yu et al., 2014), Ni (Miao et al., 2016), Mo (Krobkrong.et al., 2018), and Co (Zhang et al., 2014). Amid various TMO catalysts, Co-based catalysts showed high catalytic performance in the deoxygenation of palm oil, triolein, and stearic acid. This is attributed to the high reactivity exhibited by Co-based catalysts to convert free fatty acid feedstock into diesel-range hydrocarbon as primary products (Asikin-Mijan et al., 2018; Crawford et al., 2019; Srifa et al., 2014). Furthermore, it has been suggested that the primary role of Co-based catalysts is to promote C–C and C–O cleavage via decarboxylation/decarbonylation pathways (Asikin-Mijan *et al.*, 2018).

According to Shim et al. (2018) (Shim.J et al., 2018), the Co-based catalyst coupled with Mo metal successfully deoxygenated oleic acid and produced green diesel with 88.9% conversion and 48.1% C_9–C_{17} selectivity. Furthermore, Asikin-Mijan et al. (2018) reported that the 10 wt.% of Co metal in the (Co-Ca) supported on mesoporous SiO_2-Al_2O_3 showed a great catalytic deoxygenation reaction of triolein exclusively via decarboxylation/decarbonylation pathways by inducing the C–C cleavage and C–O cleavage and produced high hydrocarbon *n*-(C_8-C_{20}) yield of 73% (Asikin-Mijan et al., 2018). Nevertheless, Crawford et al. (2019) (Crawford et al., 2019) investigated the deoxygenation reaction of stearic acid by utilizing cobalt-based catalyst supported on zeolite NaX which achieved high conversion of stearic acid to liquid fuels 95% at 300C. While Muhammad. F.K et al. (2020) (Muhammad. F.K et al., 2020) examined the catalytic activity of Ni-Co/SBA-15 catalyst in deoxygenation of PFAD at 350 ℃, 2 h reaction time, and 10% of catalyst loading, which exhibited high deoxygenation activity with 88% hydrocarbons yield of (C_8-C_{17}) and 85% selectivity of diesel-range (C_{13}–C_{17}). Besides, the Ni-Co/SBA-15 catalyst showed good reactivity and is consistent with good stability for five runs. Consequently, the cobalt-based catalysts showed high catalytic deoxygenation activity with a superior yield of green diesel by C–C and C–O cleavage via decarboxylation/decarbonylation pathways. This is attributed to the good physicochemical properties of the catalyst, such as large number of strong acid-base sites, high crystallite size, good surface area, and pore volume (Zhang et al., 2014).

Furthermore, catalyst support plays an essential role in enhancing the dispersal of active metal sites that offers additional interaction with the reactants, which efficiently accelerate the deoxygenation reaction rate. Impressively, carbon-based support comes with a lot of benefits, some of which comprises high surface area, high thermal stability, economical and eco-friendly (Wang et al., 2018). Mesoporous activated carbon (AC) has a bright future as catalyst support due to its large pore size, which allows entering of big particle of feedstock and active metals; thus increasing the deoxygenation activity. Besides, mesoporous activated carbon (AC) has unique properties such as strong adsorption capacity, excellent mechanical properties, low affinity towards coke formation, inertness, and non-toxicity (Jain et al., 2016). Moreover, AC is thermally stable, which minimizes sintering of active metals during the deoxygenation reaction.

Several scholarly works has been reported about activated carbon (AC) development being derived from coconut shells in the manufacturing of biodiesel (Shobhana-Gnanaserkhar et al., 2020) however, till recently, the utilization of coconut-shell-derived AC in deoxygenation reactions for the production of green diesel, has not been studied vastly. Furthermore, Co-supported carbon proved its efficiency in the deoxygenation reaction of fatty acids as well as their derivatives for producing green diesel (Moreira et al., 2020). Therefore, the current work gives highlight the effectivity of several TMO (Mn, Co, Mo) alongside Ag supported over AC for PFAD deoxygenation in H_2-free conditions and solventless to produce green diesel. Indirectly, waste management from a palm oil factory also could be improved.

1.3 Hypothesis of the Research

The catalytic activity of the heterogeneous catalyst is strongly related to their surface characteristic and density of the active site. In this work, the carbon-derived coconut shell was activated with sulphuric acid/phosphoric acid to generate the acid functional groups on the carbon structure. Theoretically, a high density of acid active sites will escalate the activity of the catalysts, thus more acid sites introduced on the activated carbon surface, resulted in more catalytic activity. In practice, the green diesel is produced at 350 °C in a semi-batch reactor for several hours. Thence, in this research, the nitrogen stream with a vigorous blending of the reactant and catalyst enhanced the reaction rate of the deoxygenation process. The nitrogen flow that expelled the products out of the reactor increased the reaction rate significantly and raised the yield of the green diesel, hence reducing the time needed to complete the reaction.

1.4 Scope of Research

The wet-impregnation method utilized for the preparation of mesoporous carbon support promoted mono Co, Mo, Mn, and binary Co-Mo and Co-Ag systems, which in return manifested improvement in the physical properties of the catalysts. The mesoporous metal oxides and their mixtures supported activated carbon-derived coconut shells in exhibiting unique physicochemical properties, which reflected in their performance through the deoxygenation process. The use of mono and bimetallic catalysts supported on activated carbon was successfully assessed through the results of the deoxygenation of PFAD under a neutral atmosphere and the absence of H_2. The assessment was performed via optimization of the process parameters to find out the proper condition and the typical metal oxide concentration through series of deoxygenation experiments. The optimization of the catalysts is carried out via "one-variable-at-a time" (OVAT). Thence, the deoxygenation catalysts were characterized, the liquid and gas products were also analyzed, and the results were exhibited through tables and graphs.

1.5 Objectives of the Research

The main objective of this research is to develop a method for synthesis of activated carbon catalyst derived from coconut shells and thus doping it with transition metals by wet impregnation method to produce green diesel using palm fatty acid distillate feedstock (PFAD) via deoxygenation reaction. The aim of this study is divided into the following sections:

1. To synthesize and characterize the mesoporous activated carbon catalysts and their doping with TMOs metal (Co, Mn, Mo, and Ag).
2. To examine the catalytic performance of the synthesized catalysts and deoxygenation reaction optimization at different parameters for the production of green diesel.
3. To evaluate the reusability and stability of the synthesized catalysts in the catalytic deoxygenation of PFAD.
4. To analyze the fuel properties of green diesel in compliance with the American Society for Testing and Materials method (ASTM).

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