SYNTHESIS OF BIO-AVIATION FUEL FROM CANDLENUT (*ALEURITES MOLUCCANUS*) OIL USING SUPERCRITICAL CARBON DIOXIDE EXTRACTION

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by

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LIST OF SYMBOLS

\$	Dollar
%	Percentage
ΔG	Gibbs free energy
A	Area
adj	Adjusted
atm	Atmosphere
C	Carbon
cm	Centimeter
CO_2	Carbon dioxide
g	Gram
gal	Gallon
h	Hour
h	Planck constant
H_2	Hydrogen
HCl	Hydrochloric acid
ht	Hectare
Hz	Hertz
I	Iodine
J	Joule
K	Kelvin
K_b	Boltzmann constant
kg	Kilogram
λ	Lambda
Log	Logarithm
m	Meter

max	Maximum
min	Minimum
MJ	Megajoule
ml	Milliliter
mm	Millimeter
Mol	Mole
Mpa	Megapascal
N	Normality
^{o}C	Celsius
Pd	Palladium
ppm	Part per million
pred	Predicted
R^2	Regression coefficients
s	Second
t	Time
T	Temperature
V	Valve
V	Volume
Wt%	Percentage weight
ΔH	Enthalpy
ΔS	Entropy
μL	Microliter
Re	Rhenium

LIST OF ABBREVIATIONS

AJF	Alternative jet fuel
AOAC	Association of Official Agriculture Chemists
ASTM	American Society for Testing and Materials
ATJ-SPK	Alcohol To Jet Synthesized Paraffinic Kerosene
ATR	Attenuated total reflectance
AV	Acid Value
BAF	Bio aviation fuel
BET	Brunauer-Emmett-Teller
Bio-SPK	Bio-Derived Synthetic Paraffinic Kerosene
CCD	Central composite design
CrI	Crystallinity index
DAG	Diacylglyceride
DO	Deoxygenation
DOC	Decarbonylation
DOCX	Decarboxylation
EA	Activation energy
EDX	Energy Dispersive X-Ray
EIA	Energy Information Administration
FAMEs	Fatty acid methyl esters
FFAs	Free fatty acids
FT	Fischer-Tropsch
FTIR	Fourier Transform Infrared
FTSPK	Fischer-Tropsch synthesized paraffinic kerosene
FT-SPK/A	Fischer-Tropsch synthesized paraffinic kerosene bio-based aromatics
GC	Gas chromatography
GC-FID	Gas chromatography flame ionization detector
GCMS	Gas chromatography with mass spectrometry
GHG	Greenhouse gas
HDO	Hydrodeoxygenation
HEFA	Hydrogenated esters and fatty acids
HRJ	Hydroprocessed renewable jet

IATA	International Air Transport Association
IV	Iodine value
MAE	Microwave assisted extraction
MAG	Monoacylglyceride
NMR	Nuclear magnetic resonance
rpm	Rotation per minute
RSM	Response surface methodology
$scCO_2$	Supercritical carbon dioxide
SCQ	Sputter Coaters Quorum
SEM	Scanning Electron Microscopy
SFE	Supercritical fluid extraction
TAG	Triacylglyceride
TEM	Transmission Electron Microscopy
UAE	Ultrasound-assisted extraction
USA	United States of America
XRD	X-ray powder diffraction

LIST OF APPENDICES

SINTESIS BAHAN API BIO PENERBANGAN DARIPADA MINYAK BUAH KERAS (*ALEURITES MOLUCCANUS*) MENGGUNAKAN PENGEKSTRAKAN KARBON DIOKSIDA LAMPAU GENTING

ABSTRAK

Terdapat peningkatan minat dalam menghasilkan bahan api bio-aviation daripada tanaman yang tidak boleh dimakan untuk mengurangkan jumlah bahan api fosil yang digunakan setiap tahun. Selain itu, bahan api bio-penerbangan yang dihasilkan daripada sumber boleh diperbaharui boleh mengurangkan pencemaran alam sekitar dengan ketara dengan mengurangkan pelepasan gas rumah hijau (GHG). Kajian ini bertujuan untuk mensintesis bahan api bio-penerbangan untuk minyak kemiri sebagai bahan api alternatif. Pengekstrakan lipid daripada biji kemiri dijalankan menggunakan CO_2 superkritikal ($scCO_2$). Hasil minyak meningkat apabila tekanan $scCO_2$, suhu dan masa pengekstrakan meningkat. Reka bentuk komposit pusat (CCD) eksperimen digunakan untuk mereka bentuk keadaan eksperimen untuk pengekstrakan minyak kemiri. Menggunakan metodologi permukaan tindak balas (RSM), keadaan eksperimen telah dioptimumkan untuk pengekstrakan minyak maksimum. Pada suhu pengekstrakan $scCO_2$ 60 ^oC, tekanan 30 MPa, dan masa tindak balas 90 min, maksimum 65±0.3% minyak kemiri telah diekstrak. Model matematik Gompertz yang diubah suai telah digunakan untuk menjelaskan tingkah laku pengekstrakan lipid daripada biji kemiri menggunakan $scCO_2$. Tenaga pengaktifan positif yang diperolehi (75.39 KJ/mol) mendedahkan teknologi $scCO_2$ bergantung pada suhu pengekstrakan untuk pengekstrakan lipid daripada biji kemiri. Analisis sifat termodinamik menunjukkan bahawa pengekstrakan lipid daripada biji kemiri adalah endotermik dan tidak spontan. Sifat fizikokimia minyak candlenut menunjukkan ketumpatan 914

kg/cm^3, kelikatan 25.8 mm^2/s, takat beku -26 ^oC, nilai asid 15.8 mg KOH/g, dan asid lemak bebas (FFA) sebanyak 7.9 %. Sintesis minyak bio-penerbangan daripada lipid kemiri yang diekstrak $scCO_2$ telah dijalankan dengan kaedah hidrodeoksigenasi menggunakan mangkin Pd/ɤ-Al^{2}O^3. Pengaruh proses hidrodeoksigenasi untuk pengeluaran bahan api penerbangan bio ditentukan dengan suhu yang berbeza-beza, pemuatan mangkin, dan masa tindak balas. Selain itu, pengaruh parameter proses hidrodeoksigenasi telah dioptimumkan untuk mendapatkan hasil maksimum bahan api bio penerbangan menggunakan RSM. Hasil bahan api bio-penerbangan maksimum adalah kira-kira 90% pada keadaan percubaan optimum proses hidrodeoksigenasi: suhu 350 ^oC, pemuatan mangkin 3% berat, dan masa tindak balas 300 min. Spektroskopi inframerah transformasi Fourier (FTIR), spektroskopi resonans magnetik nuklear (NMR), dan kromatografi gas-spektrometri jisim (GCMS) bagi bahan api bio penerbangan menunjukkan bahawa proses hidrodeoksigenasi berjaya menghasilkan bahan api bio-aviation dengan nombor karbon nC8-nC17 dengan menyingkirkan kumpulan asid karboksil, ikatan rangkap karbon-karbon daripada rantai hidrokarbon. Keputusan menunjukkan bahawa bahan api bio-penerbangan boleh disintesis daripada minyak tidak boleh dimakan seperti lipid candlenut untuk mengurangkan permintaan yang semakin meningkat untuk bahan api penerbangan, meminimumkan gas rumah hijau dan meminimumkan pencemaran alam sekitar.

SYNTHESIS OF BIO-AVIATION FUEL FROM CANDLENUT (*ALEURITES MOLUCCANUS*) OIL USING SUPERCRITICAL CARBON DIOXIDE EXTRACTION

ABSTRACT

There is increasing interest in producing bio-aviation fuel from non-edible crops to reduce the amount of fossil fuels consumed annually. Moreover, bio-aviation fuel produced from renewable resources can significantly reduce environmental pollution by lowering greenhouse gas (GHG) emissions. The present study aimed to synthesize bio-aviation fuel from candlenut oil as an alternative fuel. The extraction of oil from candlenut seeds was conducted using supercritical CO_2 ($scCO_2$). The oil yield increased as the $scCO_2$ pressure, temperature, and extraction time increase. The central composite design (CCD) of experiments was used to design the experimental conditions for candlenut oil extraction. Using response surface methodology (RSM), the experimental conditions were optimised for maximum oil extraction. At a $scCO_2$ extraction temperature of 60 ^oC, pressure of 30 MPa, and 90 min reaction time, maximum of 65±0.3% of candlenut oil was extracted. The modified Gompertz mathematical model was utilized to elucidate lipids extraction behavior from candlenut seeds using $scCO_2$. The obtained positive activation energy (75.39 KJ/mol) revealed the $scCO_2$ technology depends on the extraction temperature for the oil extraction from candlenut seeds. The thermodynamics properties analyses showed that the oil extraction from candlenut seeds was endothermic and non-spontaneous. The candlenut oil physicochemical properties showed a density of 914 kg/cm^3, viscosity 25.8 mm^2/s, acid value 15.8 mg KOH/g, and free fatty acids (FFAs) of 7.9 %. The synthesis of bio-aviation oil from $scCO_2$ extracted candlenut oil was conducted with

the hydrodeoxygenation method using Pd/ɣ-Al_2O_3 catalyst. The influence of the hydrodeoxygenation process for bio-aviation fuel production was determined with varying temperatures, catalyst loading, and reaction time. Moreover, the influences of hydrodeoxygenation process parameters were optimized to obtain the maximum yield of bio-aviation fuel using RSM. The maximum bio-aviation fuel yield was about 90% at the optimal experimental conditions of the hydrodeoxygenation process: temperature of 350 ^oC, catalyst loading of 3 wt%, and reaction time of 300 min. The Fourier-transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR) spectroscopy, and gas chromatography-mass spectrometry (GCMS) analyses of bio-aviation fuel showed that the hydrodeoxygenation process successfully produced the bio-aviation fuel with the carbon number nC_8-nC_{17} by removing carboxyl acids group, carbon-carbon double bonds from the hydrocarbon chain. The results showed that the bio-aviation fuel can be synthesized from non-edible oils like candlenut oil to mitigate the rising demand for jet fuel, minimize greenhouse gas and minimize environmental pollution.

CHAPTER 1

INTRODUCTION

1.1 Background

The need for energy security and environmental concerns were the main drivers behind bioenergy growth and, consequently biofuels. Due to the fast depletion of fossil fuel resources, and environmental worries are the breaking point for energy policies to develop renewable, clean, and sustainable fuels. Nowadays, population growth has led to an increase in energy demand, especially in the aviation sector. Petro-jet fuel used to power planes must comply with strict fuel quality conditions compared to other fuels used in road transportation. The aviation industry is rapidly increasing, and the use of jet fuel was also increased following the same manner of growth, resulting in a massive negative impact on the greenhouse gas effect (Czerny et al., 2021). According to the United States Energy Information Administration, global aviation fuel usage in 2010 was over 5.2 million barrels per day. In addition, the International Air Transport Association made an aim for the aircraft industry to reduce CO_2 emissions by 50 percent by 2050. These data demonstrate the importance of integrating alternative fuels into the air travel industry.

Jet fuel is one of the petroleum fuel products that produced particularly to run airplane engines and turbines. Zhang et al. (2020) reported that according to the US Energy Information Administration (EIA) reports, jet fuel accounts for 4 gallons out of every 42 gallons of petroleum barrel. Approximately 1.5 to 1.7 billion barrels of petro-jet fuel are consumed annually by the global aviation sector (Escalante et al., 2022). The aviation industry's main operating cost is fuel, and the fluctuating price of crude oil makes long-term planning challenges. Sustainable feedstock for bio-aviation fuel would help the aviation industry become less reliant on one source of energy, avoiding crude oil fluctuating prices, and certainly minimizing greenhouse gas (GHG) emissions (Julio et al., 2021). The aviation industry is one of the most viable sectors for bioenergy. Thus, it urges immediate action to minimize greenhouse gas emissions with the rapid growth of aviation. Biofuels can provide an alternative energy source to petro fuels in the aviation industry, allowing it to decrease its carbon footprint by emissions reduction in greenhouse gases. Air travel manufacturing is seeking biofuels that are grown sustainably without any competition with land or water with food crops. Scientific endeavours in the aviation sector have been targeted towards the exploitation of plants as biofuel feedstock that are non-food plants as they contain toxic substance, fast-growing, can be planted in unproductive land that would not be utilized for food production, do not require fertilizer, irrigation, or pesticides, do not pose any risk to biodiversity, and contains high amount of oil (40-70%) (Atabani et al., 2013a). Furthermore, candlenut seeds were considered as non-food plant because the seeds can be eaten in small quantities when cooked; however, it is generally very toxic when ingested raw. It contains toxic substance and their toxicity cause abdominal pain, vomiting, and diarrhoea (Lawani & Winter 2022) In addition, these plant resources should offer a socioeconomic advantage to local communities, resulting in reducing the overall carbon cycle footprint and having equivalent or higher heating value than the existing crude oil-based traditional aviation fuel used by the air transportation sector.

From biomass feedstocks, a variety of technologies have been developed to synthesize alternative biofuels such as bio-aviation fuel, biodiesel, bioethanol, and biobutanol (Wang et al., 2021; Conteratto et al., 2021). Bio-aviation fuel is hydrocarbon with different classes such as aromatics, naphthene and paraffin ranging from C8 to C17. Bio-aviation fuel is a valuable biofuel among these renewable fuels, with greater demands on low-temperature characteristics and energy density (Goh et al., 2020).

Recently, bio-aviation fuel production from biomass resources has become a significant research field. The hydrogenated esters and fatty acids (HEFA) method was invented by Honeywell to convert vegetable oils and fats into sustainable bio-aviation and biodiesel fuel.

Hydroprocessed renewable jet fuel is presently the highly promising sustainable bio-aviation fuel that could be used to replace conventional petroleum-based fuels. Synthetic paraffinic kerosene (SPK) can be blended up to 50% with bio aviation fuel produced from biomass using the hydroprocessing method (Goh et al., 2020). The primary method for biofuel production is the transesterification of triglycerides with alcohols to produce fatty acid methyl esters, commonly known as first-generation biofuel (FAME) (Chandel et al., 2021). As it is not a "drop-in" fuel, petroleum-based jet fuel blended with biodiesel (FAME) cannot be utilized as an alternative bio-aviation fuel. Furthermore, FAME has the disadvantage of being absorbed by metal surfaces inside the engines. The highest proportion of A1 jet fuel blended with FAME of waste vegetable and Jatropha oil is 10% and 20%, respectively (Petchsoongsakul et al., 2020). These are because FAME contains oxygen, has poor cold flow characteristics, and has a lower heat content than petroleum-based fuel (Petchsoongsakul et al., 2020). As a result, many attempts to synthesize renewable and sustainable aviation fuels exist. "Hydroprocessed renewable jet fuel" (HRJ) is the next generation of biofuel, which was produced by hydroprocessing triglycerides (Elkelawy et al., 2022). This HRJ is a drop-in fuel with good cold flow properties and a high heating content. This is because HRJ is hydrocarbon-based fuel, and it is mainly n-alkanes. HRJ fuel development can decrease the amount of fossil fuels needed to meet rising liquid fuel demand while also reducing pollution, managing climate change concerns, and ensuring supply security (Wang et al., 2019). When compared to petroleum-derived aviation fuel, alternative bio-aviation fuel can substantially reduce greenhouse gas (GHG) emissions and lower the industry's carbon footprint (Kushwaha et al., 2022). Additionally, biofuels have the potential to provide additional environmental benefits, such as improved water quality, sustainability, soil quality, and biodiversity. Biofuel production can also help the global economy by increasing supply security.

Second-generation oil feedstock for production of biofuel offers several distinct advantages, including avoiding competition with existing agricultural resources, escaping the food vs. fuel debate, and being environmentally friendly and cost-effective (Ong et al, 2021). Besides, the non-edible oil possesses several desirable attributes such as biodegradability, renewability, portability, liquid nature, low aromatic content, higher heat content, and law sulfur content. Principles for defining the properties of biofuels derived from nonedible resources are grown to be a hot topic of tremendous significance as a result of the growing use of alternative fuels around the world. The existing methods for extracting the oil from nonedible feedstocks prior to bio-aviation fuel conversion are pyrolysis, soxhlet, distillation, and microwave heating (Goh et al., 2020; Ilias et al., 2022). Existing thermal-based oil extraction techniques for bio aviation fuel production have significant drawbacks, including high energy consumption, long processing times, high viscosity, and the need for additional separational processes (Goh et al., 2020; Osman et al., 2021).

Oil from various matrices have been extracted successfully using scCO_2 extraction method (Hossain et al., 2016; Hogan et al., 2021; Pattiram et al., 2022). Due to its moderate critical pressure (7.38 MPa) and low critical temperature (32 ^{o}C), CO_2 in a supercritical state is considered as an ultimate solvent for oil extraction. Additionally, CO_2 is an environmentally friendly cheap fluid, non-flammable, plentiful, and non-toxic (Hossain et al., 2016). As scCO_2 is a waterless extraction technology, the extracted oil does not require any further purification and separation process because carbon dioxide remains gas at room temperature (Ilias et al., 2022). Thus, it bears considerable interest to extract oil from candlenut seeds using $scCO_2$ for bio-aviation fuel production.

1.2 Problem statement

Human activities including fossil fuel combustion pollute the environment by emitting greenhouse gases such as nitrous oxide, carbon dioxide, and methane. The demand for air transportation fuel is rapidly increasing, with annual increases of 1.3 % predicted yearly until 2030 (Goh et al., 2020). By 2030, carbon emission from the transportation sector is estimated to increase to reach 80% compared to the current level of 24% (Solaymani. 2019) . Whereas air travel is expected to expand by 5% every year until 2026, resulting in a 3% increase in jet fuel demand (Goh et al., 2020). Conventional jet fuel is a fossil fuel product and is typically made up of C8 to C16 hydrocarbons. Commercial aviation fuel accounts for approximately 12% of total transportation energy consumption worldwide (Wei et al., 2019). Oil price fluctuations and greenhouse gas emissions are two negative consequences of fossil fuel consumption. The estimated increase in air transportation travellers and accompanying increase in fuel consumption indicates that the air travel sector requires a clean alternative fuel. The development of an alternative source, such as bio-aviation fuel, could be an effective and feasible choice, however, the high cost of bio-aviation fuel is a stumbling barrier (Goh et al., 2020). The development of bio-aviation fuel from sustainable resources would minimize reliance on fossil fuels by providing alternative sources while simultaneously lowering CO_2 emissions due to the closed cycle of carbon dioxide (Why et al., 2021). Air travel has become an essential part of modern current societal daily life. Air travel influenced global social contact, particularly in marketing and business. The enormous consumption of aviation fuel results in significant greenhouse gas (GHG) emissions, accounting for about 2% of global CO_2 emissions yearly. With increased understanding of greenhouse gas emissions, and climate change the air travel industry plans to minimize CO_2 emissions by 50 % by 2050. These concerns encourage scientists and the aviation industry to explore renewable and environmentally friendly jet fuel replacements.

There is an increasing interest on non-edible crops to use them as an alternative source for bio-aviation fuel production because of the rising demand, food vs. fuel debate, and environmental pollution concerns. Among the various non-edible crops, the candlenut contains over 60 wt.% oil (Subroto et al., 2017). So yet, no research has been done to investigate feasibility of utilizing candlenut oil for producing bio-aviation fuel. Moreover, the candlenut tree started to be commercialised in 2004 in Indonesia. The estimated area of land for cultivating candlenut tree was 210,198 ha with oil yield of 111,058 tons (Susilowati et al., 2020). The existing technology used for extracting oil from candlenut seed are mechanical extraction, soxhlet extraction, and solvent extraction. However, the existing oil extraction technologies have several limitations, including requiring prolonged extraction time, toxic chemicals, further purification and separation process. Conversely, the $scCO_2$ is an effective extraction technology. It has been extensively employed for the extraction oil from numerous substances in the food and pharmaceutical industries. The distinct advantage of this technology is that oil extracted using $scCO_2$ do not require further purification and separation process since CO_2 is gas at ambient temperature. Besides, the fluids CO_2 is an eco-friendly solvent, non-flammable, non-toxic, and cheap available solvent. Several parameters may potentially influence the $scCO_2$ extraction of oil from the candlenut. Generally, it is missing some important aspects of extraction parameters, including interaction and quadratic effects of the parameters, when the extraction experiments are conducted to evaluate the influence of one parameter at a time with the remaining other parameters constant. RSM is the combination of mathematical and statistical tools to determine the influence of parameters with considering interaction and quadratic effects among the parameters.

It is essential to determine kinetics and thermodynamics properties analyses to assess the extraction behavior of oil using a particular technology. The kinetics and thermodynamics properties analyses were conducted in the literature using linear equations. Generally, a linear equation provides the linear curve to calculate the extraction rate by considering the oil extraction data from starting value to endpoint extraction. However, the oil extraction from candlenut seeds using $scCO_2$ requires some time to interact between the oil present in the candlenut seeds and the fluid CO_2. Besides, when the oil extraction reached the maximum lipid extraction, it reached the saturated stage of oil extraction. However, the determination of oil extraction behavior using linear equations are not able to explain the lag phase and saturated phase of oil extraction. The modified Gompertz mathematical model provides a sigmoidal curve that effectively describes the lag phase, extraction phase, and saturated phase. Besides, the extraction rate calculated from the extraction phase could be further utilized to accurately describe the kinetics and thermodynamics properties of oil extraction.

The oil extracted from the candlenut seeds using $scCO_2$ contain mono and polyunsaturated fatty acids. To produce bio-aviation fuel from candlenut oil, the carboxyl acids group, carbon-carbon double, and triple bonds must be removed from the hydrocarbon chain. Hydrodeoxygenation (HDO) is an effective process to improve the H/C ratio by removing oxygen and adding hydrogen at carbon-carbon double and triple bonds. The challenge of HDO processes is that it requires extremely high pressure and an excessive amount of hydrogen gas to obtain complete removal of oxygen atoms and dehydrogenation of carbon-carbon double and triple bonds. However, studies reported that the implementation of metal oxide-based catalysts would minimize the hydrogen consumption and the deoxygenation could have occurred relatively at lower pressure. It is, therefore, in the present study, the synthesis of the bio aviation fuel was conducted with HDO process using Pd/γ-Al_2O_3 nanoparticles as a catalyst. Subsequently, the HDO process was optimized to obtain maximum aviation fuel yield using RSM. The reaction behaviour, kinetics, and thermodynamics properties are important to study the force created by and during the reaction, determining the rate of chemical reaction, and lead to better understanding of the system performance and to identify the source of energy loses during the reaction. The kinetics and thermodynamics properties of HDO process were determined using Arrhenius equations and Eyring theory, respectively. Various analytical methods were utilized to assess the successful synthesizing of bio-aviation fuel from candlenut oil. Finally, the produced biofuel properties were compared with the bio-aviation fuel specifications reported by ASTM standards.

1.3 Objectives

The main aim of this study is to synthesize bio-aviation fuel from the $scCO_2$ extracted candlenut oil. The goal of the present study was obtained with the following objectives.

i. To determine the influence of $scCO_2$ parameter, optimization, and extraction behaviour on the extraction of oil from candlenut.

ii. To identify physicochemical properties of $scCO_2$ extracted oil towards bio-aviation fuel.

iii. To synthesize the bio-aviation fuel from the $scCO_2$ extracted oil with the hydrodeoxygenation.

iv. To evaluate the physicochemical properties of bio aviation fuel towards the bio aviation fuel standard properties set by ASTM, USA.

1.4 Scope of research

This research consists of two major sections, which are candlenut oil extraction and bio-aviation fuel production. Firstly, candlenut oil will be extracted from the seeds using different extraction techniques ($scCO_2$ and soxhlet extraction). The extraction stage aims to provide the candlenut oil that will be used during this research and compare its properties with different extraction techniques. Moreover, the extraction parameters were determined to achieve the optimum oil yield. Due to the supercritical machine limitation, the extraction parameters were ranged between 32–80 ^oC for extraction temperature, 10-40 MPa for extraction pressure. Moreover, the extraction behavior was determined using Gompertz modified model. Secondly, the extracted oil was used for synthesizing bio-aviation fuel. Eliminating oxygen and cracking long carbon chain number to the bio-aviation fuel carbon range, was performed using hydrodeoxygenation process to produce bio-aviation fuel. In addition, the synthesized candlenut bio-aviation fuel was characterized to check its properties and compare it with the bio-aviation fuel ASTM standards.

1.5 Significance of research

The proposed extraction technique has many distinct advantages over other conventional extraction methods. Firstly, the $scCO_2$ extraction method is a green technology, as it uses the CO_2 gas as an extraction solvent, rather than using dangerous chemical solvents, such as n-hexane, methanol, or ethanol. Secondly, extraction time is faster than solvent extraction, and the extracted oil using $scCO_2$ have better quality than the oil extracted from other methods as it has lower acid value and FFAs content. Moreover, the separation of solvent (CO_2) from the oil is simple, as the carbon dioxide gas spontaneously boils away, leaving behind high-quality candlenut oil. Moreover, single step hydrotreatment process was used to produce bio-aviation fuel from candlenut oil. Candlenut seeds would be a valuable resource for bio aviation fuel, as it is rich with oil content and does not compete with the human food chain.

1.6 Thesis organization

This thesis has 5 chapters. Chapter 1 provides an insight into bio-aviation fuel and its resource. In addition, there was also a background study on nonedible oil extraction using the $scCO_2$ extraction method included in this chapter. The problem statement was then written after reviewing the scenario for the non-edible oil extraction and bio-aviation market. The problem statement highlights current issues in the non-edible oil extraction and bio-aviation industries, as well as the value of this research project. The aims of this research study were then carefully designed to overcome the issues that the non-edible oil extraction and bio-aviation fuel industries were experiencing. Finally, the thesis organization highlights the content of each chapter.

Chapter 2 provides an overview of various research studies in this field area that have been reported in the literature. It gives a background study on the types of non-edible oils including candlenut oil plantation of the trees and cultivation of seeds. Then, a brief view of the supercritical extraction method and its properties were also given. Furthermore, this chapter highlights the previous work done in the field of oil extraction using the supercritical carbon dioxide method. Moreover, the bio-aviation fuel resources, production pathways, and properties were also highlighted in this chapter.

Meanwhile, a design of experiment analysis was conducted to determine the most appropriate statistical methods and models for this study field.

The experimental materials and methods were presented in Chapter 3. The chapter comprise comprehensive data of the research flow as well as many experimental approaches used to accomplish it. In addition, the materials and chemicals utilized in this research were also described. This chapter also contains information that is needed for yield estimation and data analysis.

Chapter 4, some conclusions were provided in this chapter, as well as discussion of the findings from the previous study. The results and discussion chapter were divided into two parts. In the first section, the $scCO_2$ extraction parameter effect on candlenut oil extraction was determined. Moreover, in this part, the properties of the extracted candlenut oil were also described. While in the second part, the effect of bio-aviation fuel production parameters using the hydrodeoxygenation method was evaluated. Lastly, optimization studies on the $scCO_2$ extraction and bio-aviation fuel production via hydrodeoxygenation method were conducted using (RSM) to make functional interactions between the process variables and the expected response.

The last chapter in this thesis is Chapter 5, which concludes the findings achieved in this research. This chapter summarises the research findings and includes recommendations for further research in this area.

CHAPTER 2

LITERATURE REVIEW

2.1 Bio-aviation fuel feedstock

The feedstocks to produce bio-aviation fuel are various and they can be classified into three categories. The development of a sustainable aviation fuel must include consideration of the origins of the feedstock when assessing feasibility. It is possible to derive bio-aviation fuel using two types of matter that form renewable biomass, plants, and animals. Various types of biomasses, like edible oil (Wu et al., 2017), non-edible oils (Choi et al., 2015), waste cooking oils (Zhang et al., 2018), and algae (Lim et al., 2021) can be utilized as feedstock for bio-aviation fuel production. Reports indicate that about 350 crops around the world, including edible and nonedible crops could be used to produce oil and subsequently be utilized for manufacturing bio-aviation fuel (Goh et al., 2020). Bio-aviation fuel production becomes more attractive because the number of feedstock sources is relatively high. Feedstock availability is one of the most important factors in bio-aviation fuel production. There is an association between the availability of and possible yield from any cultivated feedstock. It is possible to categorize the feedstocks of bio-aviation fuel, based on the source of the biomass, into first-, second-, and third-generation biofuels. Table 2.1 illustrates which feedstocks could potentially be used in producing biofuel.

Table 2.1 Potential feedstocks for bio-aviation fuel production (Wei et al., 2019; Goh et al., 2020; Walls & Rios 2020)

First-generation feedstock	**Second generation feedstock**	**Third generation feedstock**
- Edible oil crops: palm oil, camelina, soybean, sunflower, Salicornia.	- Nonedible oil crops: Jatropha, castor.	- Algae: microalgae.
- Sugar and starchy crops: corn, sugarcane, wheat, and sugar beets.	- Municipal waste and food: used cooking oil, animal fats.	
	- Grass crops: Napier grass, switch grass.	

2.1.1 Edible oil (First generation feedstock)

Edible oil refers to any fat from sources suitable for food use. Mainly, sources from vegetable oil are used as a biofuel feedstock. Edible oil is also known as a first-generation biofuel. Using edible oil as biofuel feedstock was the most favoured at the beginning of the biofuel production era due to the accessibility of the feedstock sources and the relatively simple conversion required and a suitable substitute for biofuel (Singh et al., 2020). Currently, over 90% of total biofuel is derived from edible biomass (Ilias et al., 2021). However, the risk arises because edible oil was the primary source of the food supply, and it became a competition (Kurowska et al., 2020). High usage of edible oil as a biofuel feedstock causes an increase in the cost of food products. Furthermore, appropriate environmental conditions and limited area to grow the crops also contribute to complications in the biofuel production from edible sources of oil (Singh et al., 2020). There are ongoing debates about feedstock production in terms of whether it should become food or fuel; however, the effects have been a matter of concern since the costs of biofuels and edible oils may rise. Furthermore, in recent years, environmentalists have expressed concerns about using edible oil to produce biofuels, arguing that doing this in an extensive scale would require more edible oil crop plantations. This would deforest the areas involved and destroy ecosystems (Correa et al., 2019). Therefore, using edible oils to manufacture biofuels reinforces the debate over the optimal type of economy: food or fuel. In this respect, using edible oils to produce biofuel would compete with the production of food crops for the limited areas of potential land. Numerous countries worldwide have begun to exhibit this trend, whereby considerable areas have been used for producing oil crops so that the rising biofuel production demand can be mitigated. Ultimately, the global supply of edible oil might fall as biofuel made from edible oils replace petroleum oil fuel. Thus, these downsides became an inconvenience and shifted to further substitution of new sources for biofuel production. Table 2.2 presents a comparison of the three generations of feedstock that have been utilized for biofuel production.

Table 2.2 Advantages and limitations of biofuel feedstocks (Atabani et al., 2013a)

Biofuels	**Feedstock source**	**Advantages**	**Disadvantages**
1st generation	Edible oil	- Simple conversation process.	- Relative low oil yield. - Food vs biofuel debate. - Causes deforestation and destroys the ecosystem.
2nd generation	Non-edible oil	- Abundance availability number of non-edible crops worldwide - No debate between food vs fuel economy.	- Intractable structure of the feedstock.
3rd generation	Algal	- High oil content. - High growth rate. - Its cultivation reduces global warming.	- It requires advanced technology for biofuel conversion - It has other applications in the food, pharmaceutical, and cosmetics industries.

2.1.2 Non-edible oil (Second generation feedstock)

Although oil compositions such as fatty acids, saturated fat, and unsaturated fat in both non-edible and edible oil oils are almost similar, edible oil contains valuable nutrients and antioxidants. Conversely, non-edible oil derived from Jatropha, sea mango, rubber seed, and candlenut are not suitable for human consumption because it contains toxic substances in the oil (Atabani et al., 2013a; Singh et al., 2020). For instance, the jatropha seed oil contains purgative and curcas. Thus, oil extracted from the non-edible crops can utilize as an alternative feedstock for bio-aviation fuel production to overcome food versus fuel obstacles by tapping into non-edible oils for manufacturing biofuel. The non-edible oil plants can be grown mainly in wastelands worldwide and lessen the need for further deforestation and food supply issues (Atabani et al., 2013b). Two main factors that make feedstock consideration for producing biofuel are the percentage of oil that can be derived and agricultural harvest from the farmed land. Non-edible oils could be considered good sources of biofuel production mainly because they are easily transferable in liquid form, renewable, efficiently combustible, low sulfur and fragrance content, and biodegradable (Munir et al., 2019; Demirbas et al., 2016).

Table 2.3 shows the list of non-edible oil crops, their annual production per hectare of land per year (kg per ht per year), and percentage oil yield (wt%). The raw materials price is the main obstacle to producing biofuel. It is being reported that the raw material price for biofuel production accounts for 70–90% of the total biofuel production cost (Atabani et al., 2013b). Non-edible oil crops such as jatropha (2500 kg per ht per year) (Demirbas et al., 2016), candlenut (16000 kg per ht per year) (Pham et al., 2018), neem (2670 kg per ht per year) (Aransiola et al., 2019), karanja (900–9000 kg per ht per year) (Singh, 2020), yellow oleander (5200 kg per ht per year) (Yadav et al., 2019) sea mango (1900–2500 kg per ht per year) (Demirbas et al., 2016) grow in plenty. These plants can grow almost anywhere with minimal cultivation efforts, even in sandy and saline soils, which are not suitable for food crop production (Demirbas et al., 2016; Aransiola et al., 2019). Thus, the utilization of the non-edible oil as a feedstock would minimize the bio-aviation fuel production cost due to the cheaper raw materials source. Generally, the plantation cost for non-edible oil crops is much cheaper than for edible crops. This is because the cultivation of the edible crop requires high soil nutrition, a good irrigation system, and incentive care to maintain soil nutrients and moisture (Demirbas et al., 2016). Another important fact for determining the suitability of using non-edible oil as an alternative feedstock for biofuel production is the percentage of oil content (wt%). The percentage oil content in Jatropha seed (40–60 wt%), rubber seed (40–50 wt%), see mango seed (40–50 wt%), candlenut (60–65 wt%), polanga (60–70 wt%), and yellow oleander (60–65 wt%) (Demirbas et al., 2016; Singh et al., 2020; Pham et al., 2018; Yadav et al., 2019), which were found much higher than edible oil crops such as rapeseed (37–50 wt%), soybean (20 wt%), and palm (20 wt%) (Moazeni et al., 2019; Nongbe et al., 2017).

Table 2.3 Annual production and oil yield of non-edible oil crops

Non-edible oil crops	Scientific name	Plant type	Major crop	Yield	Oil content (wt%)	References
Jatropha	*Jatropha curcas*	tree	Seed	2500 Kg/ha/year	40-60	Demirbas et al. (2016)
Mahua	*Mahua longifolia*	Tree	Seed	20-200 Kg/ha/year	35-50	Acharya et al. (2017)
Candlenut	*Aleurites moluccanus*	Tree	Seed	80 kg/tree	60-65	Widodo et al. (2022)
Rubber	*Hevea brasiliensis*	Tree	Seed	100-150 Kg/ha/year	40-50	Atabani et al. (2013a)
Soapnut	*Sapindus mukorossi*	Tree	Seed	-	23-30	Demirbas et al. (2016)
Jojoba	*Simmondsia chinensis*	Shrub	Seed	500-5000 Kg/ha/year	40-50	Moazeni et al. (2019)
Tobacco	*Nicotiana tabacum*	Herb	Seed	1170 Kg/ha/year	35-49	Gravalos et al. (2019)
Neem	*Azadirachta indica*	Tree	Seed	2670 Kg/ha/year	25-45	Singh et al. (2020)
Karanja	*Millettia pinnata*	Tree	Seed	9000 Kg/ha/year	30-50	Pathak et al. (2019)
Castor	*Ricinus communis*	Shrub	Seed	450 Kg/ha/year	45-50	Demirbas et al. (2016)
Polanga	*Calophyllum inophyllum L.*	Tree	Seed	3700 Kg/ha/year	65-75	Demirbas et al. (2016)
Cotton	*Gossypium*	Tree	Seed	649 Kg/ha/year	17-23	Atabani et al. (2013a)
Kusum	*Carthamus tinctorius*	Tree	Seed	-	51-62	Moazeni et al. (2019)
Yellow oleander	*Cascabela thevetia*	Tree	Seed	5200 Kg/ha/year	60-65	Yadav et al. (2019)
Sea mango	*Cerbera odollam*	Tree	Seed	1900-2500 Kg/ha/year	40-50	Pathak et al. (2019)
Tung	*Vernicia fordii*	Tree	Seed	450-600 Kg/ha/year	30-40	Demirbas et al. (2016)
Bottle tree	*Brachychiton rupestris*	Tree	Seed	250-300 Kg/ha/year	50-60	Atabani et al. (2013b)

2.1.2(a) Non-edible oil crops cultivation

With rapid population growth, urbanization, and industrialization, the land area available for food production decreases. It urges proper distribution of the available land for agriculture, urbanization, commercial application, and forest reservation. If the edible oil crops are utilized as feedstocks for biofuel production, it will burden the land area available for food production. However, the non-edible crops have unique botanical features and can grow in non-fertile land like sandy, saline, and gravely soils that are not suitable for food production (Al-Maliki et al., 2021). For instance, Jatropha is a small tree or tall bush 5–7 m high. It is considered a multipurpose drought resistance tree. It can grow and survive in abandoned agricultural areas. It is a tropical tree capable of growing in different climate zones with 250–1200 mm rainfall (Atabani et al., 2013a). The jatropha tree is native to Mexico, the United States, Argentina, Paraguay, Peru, Brazil, Bolivia, and throughout the tropics, including Asia and Africa. It also has been cultivated in many harsh regions, and its yields reach about 0.5 tons per hectare in these areas (Demirbas et al., 2016).

Mahua (*Madhuca Indica*) tree is a medium-sized tree with a height of up to 20 m. It generally grows mostly in India, Pakistan, Bangladesh, and Malaysia (Acharya et al., 2017). It is a fast-growing evergreen or semi-green tree, and it can be planted in hot and wet regions. The potential production of mahua seed is about 60 MT per annum in India (Mahalingam et al., 2018). The oilseeds and oil yield in the mahua crops vary with the maturity of the mahua tree. Generally, the total oilseed yield per annum from the mature mahua tree ranges from 20 to 200 kg /ha, wherein the total oil yield per annum is about 2.7 tons per ha. The calorific value of mahua seed oil is reported to be 38.5 MJ/kg (Mahalingam et al., 2018).

Soapnut tree is generally growing in tropical and subtropical climates. The soapnut tree can grow in leached and deep loamy soils; therefore, the cultivation of the soapnut tree in leached and deep loamy soils would minimize soil erosion (Chen et al., 2012). The seeds contain 23–51.8 wt% oil, and the oil contains about 92% triglycerides (D'Ambrosio et al., 2022). Jojoba tree grows in Mexico, Mojave, and the Sonoran Deserts.38 Jojoba tree is 0.7–1.0 m high, and the jojoba fruits look like dark brown nutlike fruit. The seeds of the jojoba contain about 45 to 55 wt% lipid (Azad et al., 2019). The characteristics of jojoba oil differ fundamentally from edible oil. The chemical structure of the jojoba seeds oil contains long straight-chain ester, and the oil contains about 97% of waxed ester and 3% of FFAs (Gad et al., 2021). Jojoba oil is non-toxic, biodegradable, has a high viscosity, low volatility, and high ash points, and is relatively stable with a high dielectric constant (Azad et al., 2019). The high oil content and the wild nature of jojoba plants make it one of the best non-edible crops to be used as a potential feedstock for biofuel production.

Tobacco is one of the most common non-edible crops in the world, with enormous social and economic importance. It is an annually grown herbaceous plant widespread in South and North America, Russia, India, and Macedonia (Gravalos et al., 2019). Tobacco seed contains 35–49 wt% of tobacco oil, and the oil does not contain nicotine (Demirbas et al., 2016). The primary fatty acids in tobacco seed oil are palmitic acid, linoleic acid, stearic acid, and oleic acid. Neem is a fast-growing tree with a height of 25 m. Generally, the neem tree can tolerate high temperatures and grow in non-fertile and degraded soil (Aransiola et al., 2019). The tree originated from the Indian subcontinent but becomes a very established tree in many countries around the world, including Africa, central and south America, Bangladesh, Burma, Malaysia, Pakistan, and Sri Lanka (Demirbas et al., 2016). The fruiting of the neem tree starts at 3–5 years, but the maximum productivity of neem seeds begins after 15 years of plantation. The neem fruit has a shape that varies from oval to round, with a diameter of 1.0–1.5 cm and a length of 1.4–2.8 cm (Sushmitha et al., 2020). Neem seeds have 45 wt% oil, and it mainly contains oleic, palmitic, and stearic acids (Aransiola et al., 2019).

Karanja is a fast-growing and medium-sized leguminous tree. It can grow in various agro-climatic conditions, including clayey soil, stony soil, and sandy soil (Saini et al., 2021). The height of the karanja is about 25 m. The harvesting of the karanja seeds can be carried out after 4–6 years of plantation. The yield of karanja seeds of 0.9 to 9.0 tonnes per ha (D'Ambrosio et al., 2022). The fresh seeds contain approximately 30– 35 wt% thick yellow orange to a brown oil (Molefe et al., 2019). The karanja oil is considered a non-edible oil due to having karanjin and toxic diketone pongamol. The major fatty acids in karanja oil are palmitic, linoleic, oleic acid, and stearic acids. Castor plant grows in tropical regions worldwide, and it grows well in dry subtropical areas to wet tropics within the temperature range from 20–25 ^{o}C. The plant is drought and pest-resistant and can be grown practically where land is available. Castor seeds are poisonous to humans and animals due to the presence of ricin and other toxic compounds. The oil content in castor seeds is 46–55 wt% (Molefe et al., 2019).

Polanga is a medium to large evergreen and non-edible oilseed tree with an average length of 8–20 m. It grows on exposed sea sands or in deep soil with a 750–5000 mm per year rainfall requirement. The tree begins to yield after 4–5 years of plantation. The fruit bears a seed inside a corky shell covering and the size of the seeds is 10–20 mm (Durairaj et al., 2019). The oil yield from polanga plantation is about 2000 kg per ha per annum. Polanga seeds have a high oil content (65–75%) that contains various saturated and unsaturated fatty acids. The polanga seeds oil is greenish with thick, woodsy, or nutty smelling (Singh et al., 2020).

Kusum tree is a medium to a large-sized tree of 35 to 45 feet in height. The oil content in kusum seeds is 51–62% (Prajapati et al., 2018). The oil contains toxic cyanogenic compounds and therefore, the kusum oil is not considered edible oil. The fatty acid profile in kusum oil shows about 40% unsaturated fatty acid, and 53% saturated fatty acid (Nayak et al., 2021). Yellow oleander is a drought-resistant, and non-edible shrub. The Yellow oleander plant is native to tropics and subtropics countries and is inherent to Central and South America. The height of the yellow oleander tree is about 10–18 feet. The annual production of yellow oleander seeds is about 52 tonnes per ha, and the seeds contain 60–65 wt% oil (Atabani, et al., 2013a). Tung tree grows in native China and other countries below an altitude of 1600 m. The average height of the tung trees is about 20 m (Liu et al., 2019). The fruit's oil content is between 14–20%, the seed's oil content is between 30–40%, and the kernel oil content is between 53–60%. The average oil yield is 450–600 kg per ha per annum (Liu et al., 2019). Tung oil contains unsaturated fatty acids, a-eleostearic acid, stearic acid, and high conjugated triene fatty acid (Park et al., 2007). Moringa oleifera is a fast-growing and widely cultivated plant. Generally, the moringa plants grow in tropical and subtropical areas with a required rainfall between 250 and 2000 mm. It can grow in tolerating poor soil and dry sandy soil. The moringa seed contains 38–40% of oil, and the oil contains high-quality fatty acid (oleic acid > 70%) (Boukandoul, et al., 2018).

2.2 Candlenut oil as a bio-aviation fuel feedstock

As regards the feedstock, candlenut cultivation is currently implemented at a sufficiently large scale so to achieve a considerable production of biofuel. Due to its substantial potential as a crop, candlenut tree is anticipated to be highly sustainable. The tree can grow in poor-quality soil and in marginal areas that remain unused for standard agricultural activities as they are insufficiently productive.

2.2.1 Candlenut oil crops cultivation

Candlenut (*Aleurites moluccanus*) is also known as kemiri in Indonesia and kukui in Hawaii. The candlenut tree (*Aleurites moluccana L. Wild*) found in tropical subcontinents, belongs to the Euphorbiaceae family and is a multipurpose tree native to the Indo-Malaysia region (Pham et al., 2018). The taxonomical or scientific classification of the candlenut oil tree is presented in Table 2.4. The tree can grow in soil with a pH of 5-8 at 18-28 ^{o}C and rainfall of 6500-4000 mm and is medium sized with a maximum height of 20 m (Subroto, et al., 2017). The candlenut is a flowering tree with a crown shape, irregular branches, and large green leaves. Candlenut tree productivity reaches 80 kg of candlenut seeds per tree (Widodo et al., 2022). Indonesia currently cultivates candlenut in the eastern provinces as a perennial plant. The tree has many different uses, as almost every part can be used for various purposes. The living tree can be used as a windbreak and domestic fence (Pham et al., 2018). The wood is not resistant to rotting, but it can be used as an effective substrate for growing mushrooms, fuel, and making fishing boats. The seeds are toxic when raw, but edible when dried in small quantities. Empty seed shells can be used to make jewellery or produce dye by burning the shells. Approximately 60% of the candlenut seed is oil, which is obtained using several extraction techniques (Pham et al., 2018). Candlenut oil has a high iodine number ($\geq$125 I_2/100 g oil) and lower pour point and is primarily for cosmetics, varnishes, paints, and high-quality biofuel production. After extracting the candlenut oil, the seed cake that remains can be used as animal fodder or fertilizer. Crude candlenut oil contains high free fatty acids (FFA), contributing to its low pour point (Subroto et al., 2017). Oil content is another important criterion for determining the suitability of candlenut oil as an alternative feedstock for biofuel production (Pham et al., 2018).

Table 2.4 The taxonomical or scientific classification of candlenut oil tree

Taxonomical Classification	
Kingdom	*Plantae*
Division	*Spermatopphyta*
Subdivision	*Angiospermae*
Class	*Dicotyledoneae*
Order	*Archichlamydae*
Family	*Euphorbiaceae*
Genus	*Aleurites*
Species	*Aleurites moluccana*

2.2.2 Potential of candlenut oil for bio-aviation fuel production

The bio-aviation demand has increased due to the availability of clean sources of biofuel produced from alternative, renewable and domestic resources as well as the produced biofuels can be blended with the petro-fuel to run the existing engines (Suresh et al., 2018). As new technologies have emerged, especially in the development of alternative bio-aviation fuels, non-edible candlenut oil has been identified as a feedstock that could be used cheaply and efficiently in the future. Unlike conventional petro-fuel, biofuel is eco-friendly, and non-toxic, potentially enabling its use as an alternative for running combustion engines, thus reducing global warming and greenhouse gases emissions. Moreover, it has better lubrication properties, which prolong the life of an engine and enhance its performance. Moreover, it is secured to offer a safer and more secure way to operate since biofuel has a higher flashpoint than petro-fuel (Mahmudul et al., 2017). The cultivation of candlenut trees offers no competition to the food crops or vegetable already being cultivated. The candlenut tree has considerable potential to be utilized as a biofuel production feedstock since it is adaptable to marginal land and needs low levels of moisture and fertilizers (Siddique et al., 2011). In general, candlenut tree grows on wasteland that cannot support food crop cultivation. Thus, the food versus fuel argument could be resolved by obtaining feedstock from non-edible candlenut seeds. A further reason why the non-edible candlenut could be a suitable feedstock alternate to produce bio-aviation fuel is the distinctive characteristics of the biofuel obtained from the plant. For instance, it is biodegradable, renewable, and readily available, while its sulfur content is lower and its heat content is higher (Pham et al., 2018). A review of the literature revealed that developing a potential biofuel feedstock from candlenut plants poses a challenge to self-reliant energy security for various reasons:

i. Non-edible candlenut trees are of forest origin, it is therefore harvesting, collection and transportation are problematic.

ii. Lowering fuel economy, seasonal availability of non-edible candlenut seeds, and improper marketing channels are the major drawbacks of setting up biofuel production industries.

iii. The presence of high FFAs and moisture content requires pre-treatment to minimize FFAs and water content in the oil before the biofuel conversion process.

iv. The lack of post-harvest technologies for non-edible crops affects their oil quality.

v. Existing technologies for oil extraction and biofuel conversion are not cost-effective since these technologies require multiple purification and separation processes.

Therefore, utilizing non-edible candlenut oil as a bio-aviation fuel feedstock poses difficulties as well as opportunities, which need to be addressed to enable this alternate petro-aviation fuel to be environmentally and economically beneficial. Hence the urgency of undertaking additional studies on cultivating non-edible crops and developing biofuel conversion technology that performs cost-effectively. Its capacity to thrive in harsh, arid areas and low need for moisture mean that non-edible candlenut