EXTRACTION AND CHARACTERIZATION OF *JATROPHA CURCAS LINNAUES* SEED OIL THROUGH SOXHLET METHOD

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LIST OF ABBREVIATIONS

AEOE	Aqueous Enzymatic Oil Extraction
FAME	Fatty Acid Methyl Ester
GC-MS	Gas Chromatography-Mass Spectrometer
HAP	Hazardous Air Pollutant
MACT	Maximum Achievable Control Technology
MAE	Microwave- Assisted Extraction
PTFE	Polytetrafluoroethylene
SFE	Supercritical Fluid Extraction
TAG	Triacylglyceride
UAE	Ultrasonic-Assisted Extraction
VOC	Volatile Organic Compound

LIST OF SYMBOLS

^{0}C	Degree Celcius
h	hour
mL	mili-liter
µL	micro-liter
g	Gram
%	Percentage
min	Minute
w/w	weight of oil/weight of plant materials

PENGESTRAKAN DAN PENCIRIAN MINYAK JARAK PAGAR DARI BIJI *JATROPHA CURCAS LINNAUES* MELALUI KAEDAH SOXHLET

ABSTRAK

Penurunan pengeluaran bahan api fosil berikutan dengan susutan sumber petroleum dan keprihatinan terhadap alam sekitar telah menyebabkan dunia beralih kepada tenaga boleh diperbaharui, biodiesel dan mengurangkan kebergantugan kepada bahan api fosil. Kini, minyak jarak pagar banyak digunakan sebagai bahan mentah untuk biodiesel disebabkan harganya yang murah berbanding bahan mentah lain seperti minyak soya dan minyak bunga matahari. Kajian ini bertujuan untuk menyiasat keberkesanan pengestrakan minyak jatropha melalui kaedah soxhlet. Pengestrakan minyak jarak pagar dari biji *Jatropha curcas Linnaeus* dijalankan bawah tiga faktor utama, pelarut pengestrakan, etil asetat dan etanol, masa pengestrakan 6 jam, 8 jam, 10 jam dan nisbah pelarut kepada sampel jatropha, 7.5:1, 10:1 dan 12.5:1. Pelarut pengestrakan yang sering digunakan dalam indutsri adalah n-heksana yang disenaraikan sebagai pencemar udara berbahaya. Oleh itu, pelarut alternatif yang lebih mesra alam dan memberi keputusan akhir yang hampir dengan n-heksana telah dikaji. Parameter pengestrakan terbaik didapati melalui kajian ini telah ditetapkan berdasarkan pengeluaran minyak jarak pagar yang paling maksima. Pelarut etil asetat, masa pengestrakan 8 jam dan nisbah yang paling tinggi telah ditetapkan sebagai parameter optimum kerana memberi minyak dengan hasil yang banyak iaitu 47.38% (kg/kg). Kemudian, sampel minyak pada masa pengestrakan yang berbeza telah dianalisis melalui GC-MS untuk menilai kualiti minyak dengan menentukan konstituen minyak. Antara konstituen utama yang telah dikesan dalam minyak yang diekstrak pada masa 8 jam dan pelarut etil asetat ialah asid oleik, asid linoleik, asid palmitik, asid stearik, oleoaldehyde, metil linolate, etil oleat, metil linolelaidate, metil stearat dan squalene, di mana asid oleik dan asid linoleik adalah komponen dominan bagi kebanyakan minyak yang diekstrak. Kaedah pengestrakan soxhlet adalah lebih berkesan daripada kaedah pengestrakan lain dari segi harga, kebolehgunaan, dan juga hasil minyak dari segi kualitatif dan kuantitatif. Selain itu, etil asetat telah dipilih sebagai pelarut alternatif yang sesuai dengan kriteria mesra alam dan keberkesanan dalam memberikan hasil setanding dengan n-heksana.

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ABSTRACT

Decrease in fossil fuel production due to depletion of petroleum resources and concern toward environmental have made the world to turn into renewable energy, biofuel and reduce its dependency on fossil fuel. Jatropha oil, less expensive feedstock as it is inedible oil has been used as biofuel raw material instead of edible oils which are more expensive because of its competitive food uses. This study was carried out to investigate the performance of soxhlet extraction in the extraction of jatropha oil from *Jatropha curcas Linnaeus* seed under three main factors which were extraction solvent, ethyl acetate and ethanol, extraction time, 6 hour, 8 hour and 10 hour and solvent to solid ratio, 7.5:1, 10:1 and 12.5:1. Current extraction solvent used for jatropha oil extraction in industry is n-hexane which is listed as hazardous air pollutant (HAP). Hence, an alternative solvent which is more environmental-friendly and yields comparable result with n-hexane are studied. The best extraction condition that has been determined for maximum jatropha oil production was under solvent ethyl acetate at 8 hour extraction time and highest solvent to solid ratio. This optimum condition was finalized based on its maximum yield of jatropha oil which was 47.38% (w/w). Then, the oil samples at different extraction time were analyzed to evaluate its quality by determining its chemical constituent through GC-MS. The main components detected in the jatropha oil with solvent ethyl acetate and 8 hour extraction time were oleic acid, linoleic acid, palmitic acid, stearic acid, oleoaldehyde, methyl linolate, ethyl oleate, methyl linolelaidate, methyl stearate and squalene where oleic acid and linoleic acid are the dominant component for most of the oil recovered through extraction. Soxhlet extraction method is much more efficient than other extraction method in term of cost, operability and also yield of oil, quantitatively and qualitatively. Besides, ethyl acetate has been chosen as suitable alternative environmentally friendly as it gives comparable result with n-hexane.

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Jatropha curcas L. or its common name, physic nut, is from the family *Euphorbiaceae.* It is drought-resistant plant which grows well in marginal land of low to high rainfall areas and can be used as a commercial crop. Jatropha is easy to propagate and can easily survive with minimum care. It grows wild in many areas and even survives in infertile soil. The plant is easy to grow and produces seed for 50 years life span. The maximum oil content that has been reported for jatropha seeds has been close to 47%. However, the accepted average is 37 - 40%. Hence, it can be the best plant for future biodiesel production (Jongschaap, *et al.*, 2007). Besides using as a biodiesel feedstock, jatropha can also used for saponification of oil in process of making soap, cleaning, dye for clothes, medicinal purposes, and organic fertilizer and also as antidotes for snake bites. Jatropha oil can even be used to soften leather and lubricate machinery. Each part of jatropha plant, its leaves, flowers, fruits, and seeds has its own uses which make it a multi-beneficial plant (Gubitz, *et al.*, 1999, Felix and Clement, 2011).

Jatropha oil is a vegetable oil which is produced from *Jatropha curcas Linnaeus* (*Jatropha curcas L.)* seed. Due to the toxicity of jatropha seeds, the plant is cultivated for the sake of extracting the oil as it cannot be ingested by humans. Jatropha oil can be used as biodiesel feedstock where biodiesel means diesel fuel that is extracted from oil and fats of plants (Omotoso, *et al.*, 2011). Biodiesel is a renewable form of energy which is similar to conventional diesel fuel. Besides, this energy is environmental-friendly as jatropha oil has very low emission and it is also easily produced in rural areas.

Extraction of *Jatropha curcas L.* can be done through many methods such as ultrasonic-assisted extraction, microwave-assisted extraction, mechanical extraction, supercritical fluid extraction, aqueous enzymatic oil extraction and also soxhlet extraction (Felix & Clement, 2011). Soxhlet extraction is a method done to recover a component from either a solid or liquid (Ogbobe & Akano, 1991). The research involves extraction of *Jatropha curcas L.* seed through soxhlet method and characterization of the oil obtained through gas chromatography-mass spectrometry (GC-MS) method.

This research is mainly conducted to determine an environmental-friendly method of the extraction of *Jatropha curcas L.* seed. An optimum extraction condition which is also cost effective is required to increase the yield of the oil. The research is also to determine the constituents of the oil obtained followed by the applicability and commercialization of the oil.

1.2 Problem Statement

Recently, jatropha has been recognized as the major source of biodiesel production as it has the highest percentage of oil content which comes to an average of 37 to 40%. It has been a marked substitute in the fuel industries for automotive diesel of locomotives (Omotoso, *et al.*, 2011). According to the current ratio, the price of jatropha seeds is much lower than the oil price. But as the demand of jatropha oil as biofuel increases, the cost will increase too with consideration of jatropha plantation and the product price. Besides, the cost also depends on the quality of the oil extracted from the seed. The increasing demand of jatropha oil also has opened up wide opportunities for global marketing which leads to the requirement of competitive products which comes in advantage in term of quality, cost and production time (Openshaw, 2000). Hence, it is best to identify a best extraction technique, as to extract higher yield of oil with higher quality at lower cost.

Other than that, this research is also conducted to improve existing extraction process toward more environmental-friendly method. The existing process makes use of n-hexane, a solvent obtained from petrochemical sources which will be emitted during extraction and recovery process. Hexane is a volatile organic compound which can cause air pollution when it reacts with ozone in the atmosphere. But still, many existing processes are using n-hexane because it is lower in cost and gives good yield of oil recovery from the plant (Suzanna, *et al.*, 2003). Hence, it is necessary to come up with an alternative organic extraction solvent which gives approximately the same yield as n-hexane which is lower in cost and also environmental-friendly.

Many researches have been conducted before using jatropha and also soxhlet method, but still room for enhancement can be made. Existing extraction method can still be improved by optimizing extraction parameters such as extraction time, solvent to solid ratio and also type of solvent used. In this research, we are using ethyl acetate and ethanol as the extraction solvent replacing the current solvent hexane and study the effect of the solvent on the yield of the oil from jatropha seeds.

1.3 Objectives

The objectives of this research are

1) To study the performance of an environmental-friendly method of extraction of Jatropha curcas L. seed oil through Soxhlet method.

2) To identify the chemical composition of the oil extracted from Jatropha curcas L. seed oil through Soxhlet method using Gas Chromatography-Mass Spectrometer (GC-MS) method

1.4 Scope of Research

The scope of this research is to find the yield of jatropha oil extracted and also the efficiency of the soxhlet method. Besides that, the effect of various parameters such as extraction time, and solvent to solid ratio on the yield of jatropha oil will be studied. The extraction time used in this research will be 6 hour, 8 hour and 10 hour, where else the solvent to solid ratio will be 7.5:1, 10:1 and 12.5:1. Not only that, the effect of the type of extracting solvent such as ethyl acetate and ethanol on extraction of jatropha oil from its seed will be studied. Optimum operating condition will be obtained to maximize the yield of oil from extraction. Last but not least, the oil extracted from jatropha seed will be analyzed for its constituents using Gas Chromatography-Mass Spectrometry (GC-MS) method.

1.5 Significance of Proposed Study

This research will be helpful in determination of the performance of soxhlet method in the extraction of jatropha oil from *Jatropha curcas L.* seed based on the yield of the oil extracted. The result of this research would also signify the identification of the optimum extraction parameters for the production of jatropha oil from its seed to fulfill the high demand of jatropha oil as biofuel feedstock and also for other purposes such as medicinal use, soap making, and as natural hedge.

Besides, through this research an environmentally friendly method will be identified for the production of jatropha oil through the selection of suitable extracting solvent between ethanol and ethyl acetate. Soxhlet method has been chosen for this research as it is cheaper than other extraction method, hence reduces the production cost of the oil. This method also yields higher percentage of oil with good quality in comparison of other methods.

1.6 Conclusion

As a conclusion, Soxhlet extraction method and analysis of oil using GC-MS is one of the most reliable methods for jatropha oil extraction which is time, cost and energy saving. Despite its toxicity, jatropha oil has much benefit and has a higher prospect in the future to be commercialized in term of application of the product. Hence, an optimum extracting condition in term of time, solvent to solid ratio and extraction solvent will be identified to obtain higher yield of jatropha oil from its seed.

CHAPTER 2

LITERATURE REVIEW

2.1 An Introduction of *Jatropha curcas Linnaeus*

Jatropha is a genus of over 170 plants from the Euphorbiceaus family commonly found and utilized across most of the tropical and subtropical regions of the world. Among the different species of jatropha, *Jatropha curcas L.* has a wide range of uses and promises various significant benefits to human and industry. It has a yield per hectare of more than four times that of soybean and ten times of corn (Sepidar, *et al.,* 2009, Nobrega & Sinha, 2007). Taxonomy of *Jatropha curcas L*. is given in Table 2.1.

Table 2.1 Taxonomy of *Jatropha curcas Linnaeus*

Taxonomy	
Kingdom	*Plantae*
Division	*Embryophyta*
Class	*Spermatopsida*
Order	*Malpighiales*
Family	*Euphorbiaceae*
Genus	*Jatropha*
Species	*J. curcas*
Scientific name	*Jatropha curcas Linnaeus*
Common name	Physic nut, Barbados nut, Purging nut

(Source : Chemie, 1997)

Jatropha curcas L. is a very adaptable, perennial plant, which lives for more than two years and can be easily grown on hard soil. Jatropha plant is regarded as a shrub or small tree as its height generally ranges from 3-5 meter. It can grow in arid condition, on any kind of ground and does not require irrigation. Therefore, it can be easily cultivated in marginal land (Felix & Clement, 2011).

Growth of jatropha plant occurs during the rainy season. Flowering usually occurs during rainfall and seed will be produced at the end of the rainy season, usually in the first or second year of growth (Brittania & Lutaladio, 2010). The branches of the plant contain latex, a milky substance that hardens once out in the open air. The leaves are smooth and 10 to 15 cm in length and width. The seeds have thin shells in black colour and oblong shape. Picture of jatropha fruit and seed are given in Figure 2.1. The matured seeds are usually 2.5cm long and can be easily crushed to extract oil from them (Jongschaap, *et al.*, 2007).

Figure 2.1 Jatropha fruit and seeds

(Source : Brittania & Lutaladio, 2010)

Jatropha is believed to have been reach to countries in Africa and Asia by Portuguese seafarers from its centre of origin in Central America and Mexico via Cape Verde and Guinea Bissau. In Madagascar, Cape Verde and Benin, jatropha oil was used as mineral diesel substitute during the Second World War (Akbar, *et al.*, 2009, Agarwal, 2007).

The genus jatropha has 426 species and distributed throughout the world. *Jatropha curcas L.* was first described in 1973 by Carl Linnaeus, Swedish botanist. The name of this plant was derived from Greek where the first part of its name, ‘jatros’, meaning physician and the last part, ‘trophe’, meaning nutrition (Brittania & Lutaladio, 2010).

2.2 Jatropha Oil

Jatropha oil is a vegetable oil that is extracted from jatropha seeds of the *Jatropha curcas Linnaeus* (*Jatropha curcas L.)* plant. Jatropha oil is one of the most discussed vegetable oils that could be used as a feedstock for producing transesterified oil (biodiesel), mono alkyl esters of long chain fatty acids resulting from vegetable oil which confirm to specifications for using diesel engines. It is a clean and renewable source of fuel. The calorific value and cetane number of jatropha oil are equivalent to diesel and it can be substituted as biodiesel without any engine alteration and preheating of it (Karaj, *et al.*, 2008).

2.2.1 Chemical Properties of *Jatropha curcas Linnaeus* Oil

Chemical properties of oil are amongst the most important properties that determines the present condition of the oil. Free fatty acid (FFA) and peroxide values are valuable measures of oil quality and gives indication of the extent of deterioration of oil. The fatty acid component of jatropha oil usually contains oleic acid (41.5-48.8%), linoleic acid (34.6-44.4%), palmitic acid (10.5-13.0%) and stearic acid (2.3-2.8%) (Martinez, *et al.*, 2006). Higher iodine value of jatropha is caused by high content of unsaturated fatty acid such as oleic acid and linoleic acid (Gunstone, 2004, Akbar, *et al.*, 2009).

Jatropha oil is viscous oil, also known as heavy oil or thick oil. Viscosity is a measure of resistance of a fluid to deform under shear stress (Smalley, 2000). Due to its viscosity, jatropha oil can be made into soap, skin products, candles, perfumes and other personal care product (Sarquis & Woodward, 1999).

2.2.2 Application of *Jatropha curcas Linnaues* Oil

Jatropha oil has been found for using in many different fields for various applications around the world. There are some chemical compounds in the seed of jatropha which possesses poisonous and purgative elements and renders it from being edible to human. Therefore, jatropha oil has been commercialized in other field for non edible purposes. Some of the applications of jatropha oil which have been discussed by Felix & Clement (2011) and Parajuli (2009) have been listed below.

- Source of Biodiesel

  The extracted oil of jatropha is used as transesterified oil or biodiesel. Biodiesel is a clean and renewable energy replacing the diesel fuel. It has some advantages: it is non-flammable, non-explosive, biodegradable, and has significantly lower toxicity.

- Hedge

  Jatropha plant was commonly used as hedges or living fences to protects valuable plants from eaten by animals as it produces latex and is toxic. Besides, they also provide shade for other plants while the dropped leaves will decompose and become fertilizer.

- Fertilizer and insecticide

The residue from the oil extraction, pressed seed cake, is rich in nitrogen, phosphorous, potassium and more fertilizing nutrients. Besides this, they also have insecticidal properties which can reduce amount of nematodes in soil.

- Medicinal Uses

Tannins and latex extracted from the bark has anti microbial properties and astringent properties each. Extracts from *Jatropha curcas L.* have been shown to have anti-tumor activity, the leaves as a remedy for malaria and high fever, the seeds for treatment of constipation and the sap in accelerating wound healing procedure.

- Manufacturing

Jatropha oil gives a good foaming; white soap with positive effect on the skin is due to the glycerin content of the soap. It is also useful for manufacture of candles and cosmetic industry. The saponification of jatropha oil indicates the amount of sodium hydroxide necessary to make a solid soap. In China, it is used to make varnish while in England; it is used for wool spinning. The protein content of jatropha oil can be used as raw material for plastics and synthetic fibers.

2.3 Extraction of Jatropha Oil from *Jatropha curcas Linnaues* seed

The extraction process can be classified based on combination of phases (solid, liquid, gas, supercritical fluid). For solid – liquid, this extraction is useful for the isolation and purification of naturally occurring sources while liquid – liquid is a more common method depending on solubility properties of components.

Various solvent are used for extraction such as organic solvents and inorganic solvents where, organic solvents are less dense than water while inorganic solvents are denser than water. Commonly used organic solvents are diethyl ether, toluene, hexane, ethyl acetate, ethanol, and inorganic solvents are dichloromethane, chloroform and carbon tetrachloride.

The most oil content of jatropha is in the seed of the plant where it has about 40% of oil. The oil can be extracted using various methods as discussed below.

2.3.1 Mechanical Extraction

Mechanical pressing is the oldest and simplest method for oil extraction. No chemical is used for oil extraction. Continuous screw-presses replaced the conventional hydraulic presser equipment (Bargale, 1997). Mechanical extraction of the oil is accomplished by exerting sufficient force on confined seed. Under this condition, pressure is high enough to rupture the cells and force oil from the seed to "escape". Extraction is accomplished by compressing the material in a container that has small perforations, either round or slotted, that allow the liquid component to leave (Antony, *et al.*, 2011).

2.3.2 Ultrasonic-Assisted Extraction (UAE)

Oil extraction method based on ultrasonic waves. Ultrasound is used for the extraction of intra-cellular compounds through disintegration of cell structures which is also known as lysis of the cell (Allinger, 1975). The process of extracting oil through this method is known as cavitations. Cavitations occur when vapour bubbles of a liquid form in an area where pressure of the liquid is lower than its vapour pressure (Paula, & Roberto, 2011).

2.3.3 Microwave-Assisted Extraction (MAE)

MAE is an extraction technique which utilizes microwave energy to heat the solvent and the sample to increase the mass transfer rate of the solutes from the sample matrix into the solvent. Closed vessels are used to contain the sample and solvent, and the solvent are heated directly through the vessel where the temperature would be consistent (Manish, *et al.*, 2009).

2.3.4 Supercritical Fluid Extraction (SFE)

The basic principle of SFE is the feed material is contacted with a supercritical fluid and when the soluble materials are dissolved; the supercritical fluid containing the dissolved materials is removed from the feed material. The