
UNIVERSITI PUTRA MALAYSIA

# *EPOXIDATION OF JATROPHA METHYL ESTERS AND EVALUATION OF ITS PROPERTIES*

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# EPOXIDATION OF JATROPHA METHYL ESTERS AND EVALUATION OF ITS PROPERTIES

By

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Thesis submitted to the School of Graduate Studies,Universiti Putra Malaysia in Fulfilment of the Requirements for the Degree of Master of Science

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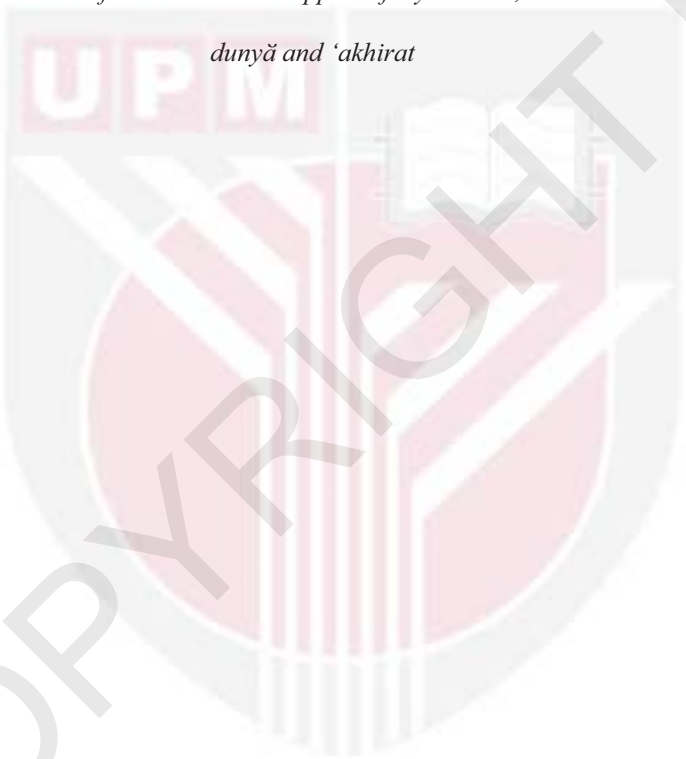
*To abah and ma*

*for their endless support of my success;*

*dunyă and 'akhirat*



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

**EPOXIDATION OF JATROPHA METHYL ESTERS AND EVALUATION OF ITS PROPERTIES**

By

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**February 2017**

**Chair: Zurina Zainal Abidin, PhD**
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Epoxides are known to have many applications in various industries because it contains highly reactive materials. Depletion of current resources is prompting researchers to find a replacement that is highly available. Jatropha seed oil is currently gaining attention because of its availability and versatility. The objectives of this study are to investigate various factors affecting production of bio-epoxides from Jatropha methyl esters (JME) through epoxidation method, to optimize production of bio-epoxides using RSM and to characterize bio-epoxides in term of physicochemical and spectroscopic characterization. JME was first synthesized using two-step transesterification method from crude Jatropha oil (CJO). Factors affecting epoxidation methods; temperature and time, molar ratio of hydrogen peroxide and acetic acid to unsaturation and catalyst loading were investigated. Physicochemical properties of JME were identified; iodine value was found out to be 107.7. Highest percentage yield of JME obtained is 90.6 with total reaction time of 4 hours. Obtained JME further used in epoxidation process. Based on RSM results, optimum temperature 70 °C, molar ratio of hydrogen peroxide 1.9 mol, molar ratio of acetic acid 0.75mol and reaction time of 3.2 hours. Maximum relative conversion to oxirane was 92.8. Spectroscopic characterization of epoxy resin was conducted using FTIR and NMR. In FTIR, unsaturation peaks were present at 3008.67 $cm^{-1}$ in CJO and JME and this peak diminished in epoxidized JME and oxirane rings surface at 825 and 843 $cm^{-1}$.$^{1}$H NMR analysis showed unsaturation peaks in between 5.27-5.32 and oxirane at 2.85 and 2.97.$^{13}$C NMR analysis presents unsaturation peaks in between 127.94-130.13 and oxirane at 56.88 ppm. In addition, blends of bio-epoxides with synthetic epoxy resin were prepared and subjected to flexural and tensile strength test. Bio-epoxides and synthetic epoxy resins blends had low tensile and flexural strength and were not suitable for applications as alternative to synthetic epoxy resin . However, it may find other application as biolubricants or polymeric coatings.

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

**PENGOKSIDAAN JATROPHA METIL ESTER DAN PENILAIAN TERHADAP SIFAT-SIFATNYA**

Oleh

**AISHAH DERAHMAN**

**Februari 2017**

**Pengerusi: Zurina Zainal Abidin, PhD**
**Fakulti: Kejuruteraan**

Resin epoksi mempunyai banyak aplikasi dalam pelbagai industri kerana ia mengandungi bahan-bahan yang sangat reaktif. Pengurangan sumber semasa mendorong penyelidik untuk mencari alternatif yang terdapat secara meluas.Minyak biji daripada pokok Jarak Pagar (CJO) kini semakin mendapat perhatian kerana ketersediaan dan serba boleh. Objektif kajian ini adalah untuk menyiasat pelbagai faktor yang mempengaruhi pengeluaran bio-epoksi daripada metil ester (JME) , mengoptimumkan penghasilan bio-epoksi menggunakan RSM dan mencirikan bio-epoksi dari segi fizikokimia dan spektroskopi dan untuk menyediakan dan menilai sifat mekanikal sintetik epoxy resin campuran resin bio-epoksi dan resin sintetik epoksi. JME terlebih dahulu disintesis menggunakan kaedah transesterifikasi daripada CJO. Faktor yang mempengaruhi kaedah pengepoksidaan; suhu dan masa, nisbah molar hidrogen peroksida dan asid asetik kepada ketaktepuan, dan pemangkin telah disiasat. Sifat fizikokimia JME telah dikenal pasti; nilai iodin didapati menjadi 107.7Hasil peratusan tertinggi JME diperolehi adalah 90.6 dengan masa tindak balas 4 jam. JME kemudiannya digunakan dalam proses pengoksidaan. Berdasarkan keputusan RSM, suhu optimum 70 °C, nisbah molar hidrogen peroksida 1.9 mol, nisbah molar asid asetik 0.75 dan tindak balas masa 3.2 jam. Penukaran relatif maksimum kepada oxirane adalah 92.8. Spektroskopi pencirian resin epoksi dijalankan menggunakan FTIR dan NMR. Dalam FTIR, puncak ketaktepuan hadir pada 3008.67 $cm^{-1}$ dalam CJO dan JME dan puncak ini tidak hadir dalam ester metil Jatropha yang dioksidakan (EJME) dan cincin oxirane wujud pada 825 dan 843 $cm^{-1}$. $^{1}H$ NMR menunjukkan puncak ketaktepuan di antara 5.27-5.32 dan oxirane pada 2.85 dan 2.97.$^{13}C$ NMR menunjukkan puncak ketaktepuan di antara 127.94-130.13 dan ikatan oxirane di 56.88 ppm. Di samping itu, bio-epoksi telah dicampurkan dengan resin epoksi sintetik, kemudian dikenakan ujian kekuatan lenturan dan tegangan. Campuran ini mempunyai kekuatan tegangan dan lenturan yang rendah dan tidak sesuai digunakan sebagai alternatif kepada sintetik epoksi resin.Walaubagaimanapun, ia boleh digunakan sebagai minyak pelincir dan salutan polimer.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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**Declaration by Members of Supervisory Committee**

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- Supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| AIER | Acidic-ion exchange resin |
| AA | Acetic acid |
| CJO | Crude Jatropha oil |
| CNMR | Carbon Nuclear Magnetic Resonance |
| $CH_3OH$ | Methanol |
| DB | Double bond |
| EJME | Epoxidized Jatropha methyl esters |
| FAME | Fatty Acid Methyl Esters |
| FFA | Free fatty acid |
| FTIR | Fourier transforms infrared spectroscopy |
| $H_2SO_4$ | Sulphuric acid |
| HNMR | Proton Nuclear Magnetic Resonance |
| HP | Hydrogen peroxide |
| JME | Jatropha methyl esters |
| MAG | Monoglyceride |
| MM | Molar mass |
| $Na_2SO_4$ | Sodium suphate |
| $CH_3NaO$ | Sodium methoxide |
| NaOH | Sodium hydroxide |
| PVC | Polyvinyl Chloride |
| RCO | Relative conversion to oxirane |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

Bioresins are defined as resins supplied from renewable sources such as plant oils and its derivatives. Bio-epoxides or also known as epoxides, are bioresins produced using epoxidation method utilizing hydrogen peroxide and peroxyacids generated *in-situ* (Cai & Wang, 2011; Goud et al., 2007; Mungroo et al., 2008). Bio-epoxides are known to have many applications in various industries because of their highly reactive materials. The main content of bio-epoxides is oxirane ring which is very reactive and enables epoxides to be used as biolubricants (Borugadda & Goud, 2015a, 2015b; Hwang & Erhan, 2001; Susana et al., 2015), reactive diluents (Das & Karak, 2009; Muturi et al., 1994), stabilizers in PVC and plasticizers (Chua et al., 2012; Witnauer et al., 1955). Besides, epoxides also have been used as intermediates in production of polyols and polyurethanes (Chen et al., 2015; Lligadas et al., 2006). This research provides brief information on epoxidation methods to produce bio-epoxides synthesized from methyl esters derived from Jatropha oil and evaluation of its mechanical properties to be used as an alternative to epoxy resins.

Currently, most available epoxy resins are synthesized from non-renewable and non-biodegradable resources such as petroleum and mineral oils. Mineral oils are important components in the synthesis of polymers known as epoxy monomer (Aouf et al., 2013), phenolic compounds (Khalil et al., 2013) and plastics (Mohanty et al., 2002) utilized for various applications in the industries. Even though the main composition of mineral oils is hydrocarbon, there are also other constituents such as sulphur, nitrogen and traces of metals which pose threat to the ecosystem due to their high toxicity. The major concern for this study was to promote the use of green technology in the chemical industries. Moreover, arising problems associated with mineral oils include depletion of reservoirs which leads to insufficient supply due to high demands and finally price increase. These problems urge researchers to find alternatives and most importantly, the new resources are preferred to be environmentally friendly in order to promote the concept of greener chemistry. A number of alternative resources have been identified such as plant oils, polysaccharide (cellulose or starch), sugar, and wood (Meier et al., 2007). All these resources are renewable and biodegradable.

This study focused on plant oils and their products of transformation because of their high availability and versatility. Plant oils mainly composed of mixtures of triacylglycerol, diacylgycerol, monoacylglycerol and free fatty acid at different percentages (Salimon et al., 2014). Physiochemical properties of plants oils depend on the composition of fatty acid content. The presence of unsaturation or double bonds

indicated by their iodine value (IV) contributes to unique chemical structures that allow chemical modifications to improve their characteristics.

Epoxidation is one of the modification methods to produce biopolymer from plant oils and its derivatives. It is described as the formation of an oxirane group by the reaction of peroxyacids and aromatic double bonds. There are a few factors that affect epoxidation reaction and must be observed carefully, including methods of epoxidation, properties of raw materials, types of epoxidation reagents and solvents, and the presence and type of catalyst (Okieimen et al., 2002). Basically this process is conducted with the presence of organic acid (formic or acetic acid), hydrogen peroxide and catalysts (Campanella et al., 2008; Mushtaq et al., 2013). Organic acids react with hydrogen peroxide to produce peroxyacids, either *in-situ* formed or preformed peroxyacids, with or without presence of catalysts. As a matter of fact, hydrogen peroxide is regarded as a very powerful oxidizing agent which may lead to spontaneous combustion when come in contact with organic material. Concentrated peroxyacids are also unstable and may cause explosion. Because of safety concerns, *in-situ* formed peroxyacids process is a preferable method compare to preformed peroxyacids (Goud et al., 2007).

Jatropha *curcas L*. are parts of Euphorbiaceae family and originated from South America and Africa. However, it also can be found in South East Asia and India. It is a small tree with large shrub. Their size is between 5 m height and at certain conditions; the height can reach to 8 to 10 m (Insanu et al., 2013). *Jatropha curcas* seed oil was chosen as the feedstock material in this study. Jatropha oil can be extracted from leaves, fruits or seed of the plant, shown in Figure 1.1. The seeds of Jatropha plant are toxic to animals and human. Thus, the oil is not fit for consumption or any other applications in food industry. Hence, production of FAME from crude oil increases the economic value as well as productivity of the oil.

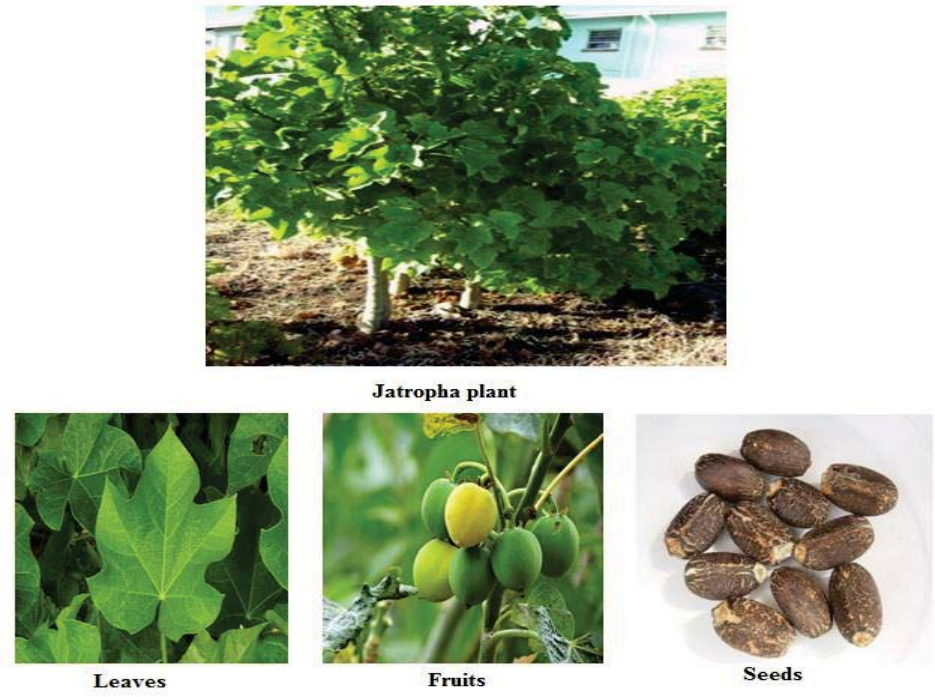


**Figure 1.1: Different parts of Jatropha plants**

In this paper, we report synthesis of bio-epoxides from FAME derived from crude Jatropha oil using the epoxidation method. The process was initiated by the production of methyl esters from crude Jatropha oil by employing transesterification method whereby acid and alkali were used as catalyst. Optimization of epoxidation was obtained using response surface methodology (RSM).

## 1.2 Problem Statements

Jatropha methyl esters (JME) have high iodine value which makes them suitable to be used as raw material in the production of bioepoxides. Epoxidized Jatropha methyl esters (EJME) has high oxirane value, therefore has an excellent potential to be used as bio-epoxy resins due to high reactivity of oxirane ring. Previous works has synthesized bio-epoxides from JME using conventional epoxidation methods (Mushtaq et al., 2013; Campanella et. al., 2008). Other works by Borugadda & Goud, (2014) also produces bio epoxides from Castor oil methyl esters using heterogeneous catalyst like ion acidic exchange resin (AIER). Epoxidation method using AIER is an effective method because it minimizes side reaction, high selectivity to epoxidized product and it is a clean and environmental- friendly catalyst. Optimization of production bio epoxy resin from JME is important to find out the optimum operating conditions to maximize production of bio-epoxides. Current epoxy resins are synthesized from petroleum resources. The depletion

of the resources urges the need to find alternative resources that available in abundant. Moreover, the usage of synthetic epoxy resin derives from petroleum raises many issues in terms of safety of use towards health and environmental impact. Bioresources derived from plant oil is an excellent substitutes because besides being available in abundant, they are also environmental friendly. No work has ben done to synthesize bio-epoxides from JME using AIER method. Therefore, this work was conducted to optimize the production of bio-epoxides from JME using AIER epoxidation method and spcectroscopically characterize it. Bio-epoxides obtained from epoxidation process was blended with synthetic epoxy resins and subjected to tensile and flexural test.This is important to asses the properties of bio-epoxides in comparison to synthetic epoxy for further applications. Bio-epoxides produced through epoxidation of JME have potential as plasticizer and stabilizer in PVC, biolubricants and as a polymeric coating.

## 1.3 Research Objectives

The main purpose of this study was to investigate the synthesis of Jatropha methyl ester using acidic ion exchange resin (AIER) as catalyst.
The objectives of this research are:

- To investigate various factors affecting production of bio-epoxides from JME through epoxidation method and optimize the production of bio-epoxides using response surface methodology (RSM).
- To characterize bio-epoxides in term of physicochemical and spectroscopic characterization

## 1.4 Scope of works

i. This study focus on production of bio-epoxides from JME through epoxidation method. During preliminary studies, five reaction parameters were investigated namely, reaction temperature, time, molar ratio of hydrogen peroxide to unsaturated carbon, molar ratio of acetic acid to unsaturated carbon and percentage weight of catalyst loading. The range of investigated parameters were; temperature (30-85˚C) molar ratio of hydrogen peroxide to unsaturated carbon (0.8-2.5 mol), molar ratio of acetic acid to unsaturated carbon (0.3-1.0 mol), catalyst loading (5-20%) and time (1-10 hours).

ii. Different from previous works, this work focused on epoxidation of JME using combination of peroxyacetic acid generated *in-situ* and Amberlite-120 catalyst with the purpose to accelarate the formation of epoxides and reduce the usage of hydrogen peroxide. Optimization study was conducted to investigate the optimum reaction parameters and Response Surface Methodology (RSM) method was used. Central composite desing (CCD) was adopted and four reaction parameters were studied which were temperature (30-85˚C), molar ratio of hydrogen peroxide to unsaturated carbon (1.1-2.5 mol), molar ratio of

acetic acid to unsaturated carbon (0.5-0.8mol) and reaction time (2-7 hours).The results were analysed using ANOVA.

iii. Physicochemical JME and epoxidized Jatropha methyl esters (EJME) such as iodine value, acid value, free fatty acid, saponification value, density, viscosity and specific gravity were identified. GC-MS analysis were done to evaluate the fatty acid compositions of JME. Spectroscopic characterization, FTIR and NMR were used to identified molecular structure in both JME and EJME. Hence, to confirm formation of bio-epoxides. Evaluation of mechanical properties; tensile and flexural strength of bio-epoxides and synthetics epoxy resins blends were conducted to evaluate the suitability of bio-epoxides as an alternative to synthetic bio-resin.

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