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SYNTHESIS AND CHARACTERIZATION OF EPOXIDISED JATROPHA OIL FOR BIO-EPOXY RESINS

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By

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SYNTHESIS AND CHARACTERIZATION OF EPOXIDISED JATROPHA OIL FOR BIO-EPOXY RESINS

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Synthetic resin has been used in various applications such as an adhesive and in laminating, coating, and painting. Despite its wide applications, its usage raises issues for health, environmental and safety concerns. The synthetisation of bio-resin from renewable resources will be able to introduce compostable and biodegradable plastics which can directly solve many issues such as the floating of the non-biodegradable plastics in the oceans and health concerns of the usage of bisphenol-A. Studies have shown that bio-resin can be used as a bio-reinforcement in a composite formulation. However, there is no research done to produce a hybrid bio-matrix for composite application by improving its formulation of blended synthetic resin with bio-resin. Thus, this study would like to introduce the optimisation and characterisation of the epoxidation reaction of Malaysian crude jatropha oil with the presence of ion exchange resin as well as the mechanical properties of the hybrid bio-matrix. The study found that the optimum epoxidised crude jatropha oil (ECJO) was achieved by reacting 1 mol (263.16 g) of crude Jatropha oil with 0.6 molar ratio of glacial acetic acid, 1.7 molar ratio of 30% hydrogen peroxide, and 16 wt. % of ion exchange resins. The optimal epoxidation process was carried out at a temperature of 60 °C for 5 hours. The epoxidation process was accomplished by obtaining a high oxirane content (OOC) of 3.71%. The formation of oxirane ring was confirmed by FTIR, ^{13}CNMR, and ^{1}HNMR at 822 cm^{-1}, 54.203-57.26 ppm, and 2.809-3.031 ppm respectively. The activation energy (E_a), average activation enthalpy (ΔH), and average activation Gibbs free energy (ΔG) of the epoxidation process were found to be 35.91 kJ/mol, 33.16 kJ/mol, and 117.21 kJ/mol respectively. The hybrid bio-matrix with the blend of 25-75% of the ECJO\Epoxamite 103 was found to be relatively rigid and brittle with the tensile strength and modulus as well as flexural strength and modulus of 30.29±4.43 MPa, 464.46±137.10 MPa, 47.44±2.57 MPa, and 1568.59±60.61 MPa respectively. Thus, it is recommended to use 10-25% of the epoxy bio-resin in blends with the standard commercial epoxy resins as an optimal replacement for the synthetic petro-based products.

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

SINTESIS DAN PENCIRIAN EPOKSI MINYAK JATROPHA UNTUK BIO-EPOKSI RESIN

Oleh

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Sintetik resin telah digunakan dalam pelbagai aplikasi seperti pelekat, lapisan, salutan, dan cat. Walaupun aplikasinya meluas, penggunaannya telah menimbulkan pelbagai isu dari segi kesihatan, alam sekitar, dan keselamatan. Sintesis bio resin daripada sumber yang boleh diperbaharui mampu memperkenalkan satu plastik yang boleh dikompos dan terurai dimana secara tidak langsung boleh menyelesaikan banyak isu-isu seperti apungan plastik yang tidak terurai di lautan dan kebimbangan penggunaan Bisphenol-A. Kajian telah menunjukkan bahawa bioresin boleh digunakan sebagai bio-pengukuhan dalam penggubalan komposit. Walau bagaimanapun, tidak ada penyelidikan yang dilakukan untuk menghasilkan hibrid bio-matriks dalam pembuatan komposit dengan meperbaiki formula campuran dengan mencampurkan sintetik resin dengan bio-resin. Oleh itu, kajian ini ingin memperkenalkan pengepoksidaan dan pencirian secara optimum oleh minyak jatropha mentah Malaysia dengan kehadiran ion pertukaran resin serta sifat-sifat mekanikal hibrid bio-matriks. Kajian mendapati bahawa optimum epoksi minyak jatropha mentah (ECJO) telah dicapai dengan bertindak balas 1 mol (263.16 g) minyak Jatropha dengan 0.6 nisbah molar asid asetik glasier, 1.7 nisbah molar 30% hidrogen peroksida dan 16 wt. % ion pertukaran resin. Proses ini telah dijalankan pada suhu 60 °C selama 5 jam. Proses pengepoksidaan dicapai dengan kandungan oxirane yang tinggi (OOC) iaitu sebanyak 3.71%. Pembentukan cincin oxirane telah disahkan oleh FTIR, ^{13}CNMR, dan ^{1}HNMR di 822 cm^{-1}, 54.203-57.26 ppm, dan 2.809-3.031 ppm. Tenaga pengaktifan (E_a), purata entalpi pengaktifan (ΔH), dan pengaktifan purata Gibbs tenaga bebas (ΔG) oleh proses pengepoksidaan yang didapati adalah sebanyak 35.91kJ/mol, 33.16 kJ/mol, dan 117.21 kJ/mol. Matriks hibrid bio dengan campuran 25-75% daripada ECJO\Epoxamite 103 didapati agak tegar dan rapuh dengan kekuatan tegangan dan modulus serta kekuatan lenturan dan modulus sebanyak 30.29±4.43 MPa, 464.46±137.10 MPa, 47.44±2.57 MPa, dan 1568.59±60.61 MPa. Oleh itu, adalah disyorkan untuk menggunakan 10-25% bio-epoksi resin dalam campuran dengan epoksi resin komersial sebagai pengganti optimum produk berasaskan petro-sintetik.

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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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LIST OF ABBREVIATIONS

Abbreviations	Definition
%RCO	Percentage Relative Conversion to Oxirane
AIER	Acidic Ion Exchange Resins
AOCS	American Oil Chemist's Society
ASTM	American Society for Testing and Materials
BPA	Bisphenol-A
^{13}CNMR	Carbon-13 Nuclear Magnetic Resonance
CJO	Crude Jatropha Oil
COA	Certificate of Analysis
DB	Double Bond
DGEBA	Diglycidyl Ether of Bisphenol A
ECJO	Epoxidised Crude Jatropha Oil
EEW	Epoxy Equivalent Weight
FFA	Free Fatty Acids
FTIR	Fourier Transform Infrared Spectroscopy
^{1}HNMR	Proton Nuclear Magnetic Resonance
IV	Iodine Value
LHHW	Langmuir-Hinshelwood-Hougen-Watson
MM	Molar Mass
MSDS	Material Safety Data Sheet
OO	Oxirane Oxygen
OOC	Oxirane Oxygen Content
TAG	Triglyceride

GLOSSARY OF TERMS

Notation	Definition
A	Temperature Independent Factor
B	Concentration of Hydrogen Peroxide (mol/L)
Bf	Final Concentration of Hydrogen Peroxide (mol/L)
C_{aao}	Initial Concentration of Acetic Acids (mol/L)
C_{ep}	Concentration of epoxides (mol/L)
C_{hpo} or Bo	Initial Concentration of Hydrogen Peroxide (mol/L)
cP	Centipoise
cSt	Centistokes
E_a	Activation Energy
h	Planck's Constant (6.626×10^{-34} J/s)
k	Rate Constant (L/mol.s)
MPa	Mega Pascal
N	Avogadro's Number (6.022×10^{23} mol^{-1})
ppm	Parts per million
R	Gas constant (8.314 kJ/mol.K)
R^2	Coefficient of Determination
T	Absolute Temperature (Kelvin)
Wt. %	Weight Percentage
ΔG	Activation Gibbs Free Energy (kJ/mol)
ΔH	Activation enthalpy (J/mol)
ΔS	Activation Entropy (J/mol.K)

CHAPTER 1

INTRODUCTION

1.1 Background

Resins can be defined as solid or semi-solid compounds which consist of organic materials. Natural resins can be found in the trees when they exude some liquids to cover the wounded parts due to tapping incident. Synthetic resins can be defined as resins that have been synthesised through chemical synthesis. On the other hand, bio-resins are resins that have been synthesised by using renewable resources such as vegetable oils, lignin, cellulose, starch, chitin, and chitosan (Gandini & Lacerda, 2015).

One of the most evolving thermoset polymers is epoxy resins. As soon as epoxy resins have been introduced in industries, it has been commercialised in various applications namely as coating, painting, laminating, flooring, and paving. Epoxy resins are commonly used as matrices in fabricating composite (Benyahya et al., 2014). Hence, epoxy resins also play important roles in construction of bicycle, automobiles, aircraft, boats, skis, and snowboard (Jin et al., 2015).

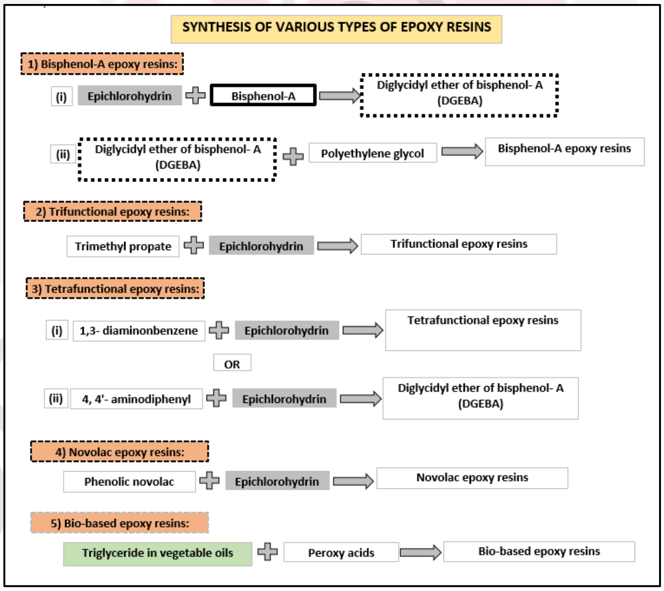

Figure 1.1: Synthesis of various types of epoxy resins (Adapted from Jin et al., 2015)

Generally, there are two types of epoxy resins which are synthetic epoxy resins and bio-based epoxy resins. Synthetic epoxy resins are widely used in construction industries for impregnation, casting, gluing, and flooring (Mateusz et al., 2015). The most well-known petroleum-based products in synthesising the synthetic resins are epichlorohydrin and diglycidyl ether of bisphenol-A (DGEBA) as shown in Figure 1.1. DGEBA is commonly produced by reacting epichlorohydrin with the bisphenol-A (BPA) (Jin et al., 2015). DGEBA has been reported as the best epoxy component in the thermosetting polymer due to its aromatic structure which allows the overall performance to the cured resins (Pawar et al., 2016). On the other hand, epichlorohydrin is mainly produced from petroleum-based products (Pawar et al., 2016).

However, the usage of the bisphenol-A in the production of DGEBA has been reported to give many harmful effects to the human health to the extent that many industries are restricted the usage of BPA especially in the food contact products such as baby bottles (Pawar et al., 2016). Toxicity of BPA, high cost of fossil fuel, and depletion of the petroleum resources have caused many chemists in the industries to find alternative resources for replacement of petroleum-based source of resin.

Recently, many researchers have introduced various types of renewable resources as the starting materials in the production of epoxy resins (Ferri et al., 2011). It has been reported that the renewable resources such as vegetable oils, lignin, cellulose, starch, terpenes, as well as derivatives of the microbial activity (Gandini & Lacerda, 2015; Ronda et al., 2013) are suitable to be used as the starting material in synthesising various types of resins namely alkyd resins, polyesteramide resins, polyetheramide resins, polyurethane resins, epoxy resins, and polyol resins (Alam et al., 2014).

For the past decades, many researchers have studied various types of vegetable oils such as sunflower oil (Martín-Alfonso & Franco, 2014), castor oil (Borugadda & Goud, 2014; Shoaib et al., 2014), jojoba oil (Shoaib et al., 2014), and corn oil (Mustata et al., 2014) in epoxides production due to the versatility of the constituents of vegetable oils.

The methodology of the epoxidation in epoxides production has been investigated by many researchers which can be categorised into four types namely the conventional epoxidation method (by using strong acid as catalyst), the chemo-enzymatic epoxidation method (by using enzyme as catalyst), the metal-catalysed epoxidation method (by using metal as catalyst), and the ion exchange resin method (by using ion exchange resins as catalyst) (Manthey et al., 2010; Salimon et al., 2014; Saurabh et al., 2011). Epoxidation by using ion exchange resins has been reported to be the most effective in producing high concentrations of oxirane ring in the products due to the behaviour of ion exchange resins which act as heterogeneous catalysts (Manthey et al., 2010).

Therefore, this work was intended to optimise the epoxidation of Malaysia crude jatropha oil (CJO) in the presence of ion exchange resins, Amberlite IR-120 as a heterogeneous catalyst. Further work was also done on the characterisation of the epoxidised crude jatropha oil (ECJO) followed by the kinetics and thermodynamics study of the epoxidation process. In addition to that, the mechanical properties of the blended epoxidised crude jatropha oil (ECJO) with the commercial synthetic epoxy resins were also determined.

1.2 Problem Statement

Synthetic resin is commonly produced from petrol-chemical products through the chemical synthesis. Although it has been used in various applications, the usage of synthetic resin raises many issues in terms of safety of use towards health and environmental impact. Many efforts have been done in producing bio-resin by taking various plants-products as the starting materials such as crops, agricultural waste, vegetable oils, and cellulose. Among all the resources, vegetable oil is the most preferable starting material since it is a huge resource and traded on a global basis. The idea of producing bio-epoxy resins through the epoxidation of vegetable oil is to replace the petroleum-based content in the conventional synthetic resins with renewable and sustainable resources. The usage of bio-resins is directed into various range of products and packaging namely in food service, personal care, bags, and cosmetic packaging. Since vegetable oil is a renewable resource, the resultant synthesised bio-resins will be able to introduce compostable and biodegradable plastics. The toxicity of phorbol esters presence in crude jatropha oil prevents its usage in the food industry, however, that consequently made it as the best starting material for production of epoxy resin through epoxidation reaction with the presence of an ion exchange resin as a heterogeneous catalyst. The catalyst would provide a simple separation process of acid from epoxides, low degradation of oxirane ring, and a green epoxidation process since the catalyst is environmentally friendly. Studies have showed that bio-resin can be used as a reinforcement in a composite formulation. However, only a little research is done to produce a hybrid bio-matrix for composite application by replacing a certain amount of the synthetic resin with the bio-resin. The blended matrix will provide good opportunities in adjusting the flexibility of composite and good potential in producing a genuine bio-composite material by incorporating the hybrid matrix with the bio-reinforcement. Therefore, this study is proposed to optimise and evaluate the kinetics of the epoxidation crude jatropha oil with the presence of Amberlite IR-120. Further physico-chemical and spectroscopic characterisation of the epoxidised crude jatropha oil as well as mechanical characterisation of the hybrid matrices obtained between the bio-epoxy resin and synthetic resin were also determined.

1.3 Objectives

There are 4 main objectives of this research which are:

i. To optimise the epoxidation reaction of the crude jatropha oil.
ii. To characterise the epoxidised crude jatropha oil (ECJO).
iii. To evaluate the kinetics and thermodynamics activation energy of the epoxidation process.
iv. To determine the mechanical properties of the hybrid matrix by blended epoxidised crude jatropha oil (ECJO) with the synthetic resin (Epoxamite resin).

1.4 Scope of Research

1. This study emphasised on the vegetable oils located only in Malaysia as the starting materials for epoxidation process with focusing on the iodine value (IV) property. The optimisation was done based on one factor at a time with the variables of temperature, molar concentration ratio of acetic acid to ethylenic unsaturation carbon, molar concentration ratio of 30% hydrogen peroxide to ethylenic unsaturation carbon, and weight of catalyst (Amberlite

IR-120) in the range of 30-80 °C, 0.3-1.2 mol, 0.8-2.5 mol, and 5-20 wt. % respectively.

2. The epoxidised crude jatropha oil (ECJO) was characterised in terms of FTIR and NMR spectroscopy as well as physico-chemical properties.
3. The kinetics study was evaluated by using pseudo-homogeneous model at the first order by considering the Amberlite IR-120 acted as homogeneous catalyst since there was no swelling of the catalyst throughout the reaction. The thermodynamic study was evaluated in terms of activation enthalpy, entropy, and Gibbs free energy.
4. The mechanical properties of the cured epoxidised crude jatropha oil (ECJO) blended with the synthetic resins were identified in terms of tensile and flexural strength as well as modulus.

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