Optimization in Iodine Value for Mixture of Edible and Non-Edible Oil for Epoxidation Process

by

Tan Seng Yee

Dissertation submitted in partial fulfillment of

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Bachelor of Engineering (Hons)

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CERTIFICATION OF APPROVAL

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Approved by,

_____

(Dr. Ruzaimah Nik Mohamad Kamil)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH PERAK

May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the reference and acknowledgements, and that the original work contained herein have not be undertaken or done by unspecified sources or persons.

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TAN SENG YEE

ABSTRACT

The study of optimization of Iodine Value (IV) for the mixture of edible and non-edible oil for epoxidation process has been carried out in this project. Edible oil which referred to palm oil in this project has a lower IV of 55g/100g oil as compared to other types of oil such as rubber seed oil or Jatropha oil. Lower IV from palm oil will result in a lower yield of percentage oxirane oxygen content (%OO), approximately 3 to 3.5 depending on the type of palm oil used. Mixing palm oil with different types of non-edible oils with higher IV such as Jatropha oil and rubber seed oil at different weight ratio was done in order to increase the IV of the oil mixture made. Testing of the IV was carried out on all samples from different mixing ratios. It was found out that the weight ratios of oil mixtures and their respective IV is in a linear relationship. The optimum weight ratio in order to obtain desired amount of IV and $\%OO_{TH}$ has been identified based on previous study which is 25 wt% of palm oil and 75 wt% of rubber seed oil.

ACKNOWLEDGEMENT

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ABBREVIATIONS AND NOMENCLATURES

EJO	Epoxidized Jatropha Oil
EPO	Epoxidized Palm Oil
ERSO	Epoxidized Rubberseed Oil
IV	Iodine Value
PO	Palm Oil
%OO	Percentage Oxirane Oxygen
RSM	Response Surface Methodology
$\%OO_{TH}$	Theoretical Percentage Oxirane Oxygen

CHAPTER 1

INTRODUCTION

1.1 Background

The depletion of petroleum-based material such as petroleum oil is being concerned in the future since it is not renewable and will deplete if no other alternatives are used to substitute petroleum-based materials. Substitution of petroleum based-material by vegetable oil-based materials is being paid attention widely as vegetable oils are renewable resources that are environmentally friendly, biodegradable, low cost and readily available. This application will help to preserve the world by reducing the global warming affect since environmental friendly and biodegradable materials are used. Application of vegetable oil-based materials in petroleum based-materials can be done by applying epoxidation process.

Epoxidation process is very common nowadays and it has been applied for karanja, mahua oil, vegetable oil, soybean oil, Jatropha oil, rapeseed oil, cottonseed oil, and canola oil nowadays. Homogenous or heterogeneous catalysts are normally added in the reaction for a higher yield of percentage oxirane oxygen (%OO) Choosing heterogeneous catalyst can be more advantageous when it comes to the separation of catalysts and reaction product. Heterogeneous catalyst such as ion exchange resin is always being chosen to be used in epoxidation process because side reaction from epoxidation can be minimized and thus maximizing the selectivity of the reaction.

Epoxidation process to produce Epoxidized Palm Oil (EPO) has been carried out and the optimization operating conditions were identified by Malaysia Palm Oil Board (MPOB) in 2009. However, the low IV in palm oil has leaded to a lower

amount of %OO produced from the epoxidation process as compared to other types of oils such as Jatropha oil and rubberseed oil. Optimization of IV in raw materials has to be paid extra attention for a better yield in epoxides group at the epoxidized oil. In this project, it is being proposed that mixing between edible oil and non-edible oils at different weight ratio can be done to produce an oil mixture with higher IV.

1.2 Problem Statement

Epoxidation process is a technology that attracts worldwide attentions nowadays, considering on its effective way in keeping a greener environment. Most of the vegetable oils such as cottonseed oil or soybean oil are normally selected for epoxidation process due to their higher iodine value (IV) or normally referred to the number of double bonds in the particular vegetable oils. These optimization studies had been conducted for various types of vegetable oils synthesized by the epoxidation process using different type of catalysts.

The optimization study for the synthesis of EPO has been done as well in order to get the highest yield of epoxides. Palm oil is selected in the study because palm oil is widely available in Asian countries such as Malaysia or Thailand. Despite of its lower IV as compared to other vegetable oils, palm oil is selected due to its wide availability. The problem faced by EPO is mainly on the highest yield of epoxides that can be produced is still relatively low as compared to other type of oil. Optimum condition to produce EPO has been identified and methods to slow down the rate of degradation of epoxides are being figured out recently by using antioxidant. However, attention should be paid on the optimization of IV in palm oil itself because palm oil has lower IV or amount of double bonds.

In this project, palm oil which is a type of edible oil will be mixed with two types of non-edible oil which are Jatropha oil and rubber seed oil. Three types of oil will be mixed at different weight ratio to produce a new mixture of oil. The IV of new mixture of oil will be tested and the data will be collected. The mixture of oil that gives out the desired amount of IV will be used to undergo epoxidation process. This approach can provide alternative to allow palm oil to be used in epoxidation process and yet produce a good yield of epoxides.

Problem Statement: What is the optimum weight ratio for the mixture of edible oil (palm oil) and non-edible oil (Jatropha oil and rubber seed oil) to optimize the iodine value (IV) for epoxidation process?

1.3 Objective and Scopes of Study

The main objective of the study is:

- To investigate the optimum weight ratio for the mixture of edible oil (palm oil) and non-edible oil (Jatropha oil and rubber seed oil) to optimize the iodine value (IV) for epoxidation process.

In order to obtain a better result in term of accuracy and precision, the scope of the study has been narrowed down to a certain point in which the whole study will be conducted. This can ensure that the project conducted can meet all the expectation and objectives identified before. The scopes that are identified for this project are as follows:

a. The study of optimization in IV will be carried out with only one type of edible oil which is palm oil and two type of non-edible oil which are Jatropha oil and rubber seed oil.
b. The combination of the mixing of oil will be made between palm oil and either with Jatropha oil or rubber seed oil as well as palm oil with Jatropha oil and rubber seed oil.
c. Selection of the oil mixture used for the study in optimization of epoxidation will be made between mixing of palm oil and rubber seed oil only.

CHAPTER 2

LITERATURE REVIEW/ THEORY

2.1 Fatty Acid

Fatty acid is a carboxylic acid with long aliphatic chain. These fatty acids are commonly present in animal fats such as duck fat or butter or vegetable fats such as coconut oil and olive oil. It is commonly being classified into two major types of fatty acids which are saturated and unsaturated fatty acids. Saturated fatty acids can be defined as fatty acids that have no double bond and have the maximum number of hydrogen bonded to the carbons. Figure 2-1 shows one of the examples of saturated fatty acids, stearic acid ($C_{18:0}$). It does not have any carbon-carbon double bond in the chemical structure.

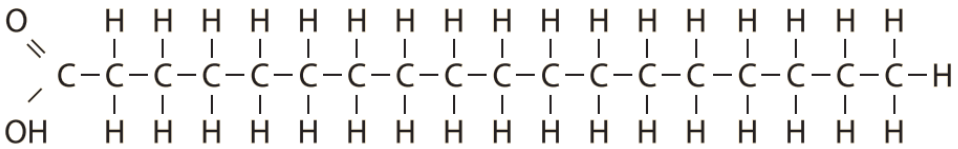

Figure 2-1 Saturated fatty acids: stearic acid

Unsaturated fatty acids can be defined as fatty acids in which there is at least one double bond within the fatty acid chain. Unsaturated fatty acids can be converted into saturated fatty acids by adding hydrogen atoms to them, converting the double bonds to single bonds. They will be defined as monounsaturated fatty acids if there is only one double bond within the fatty acid chain.Figure 2-2 shows example of

monounsaturated fatty acids in palm oil, oleic acid ($C_{18:1}$) that has only one double bond.

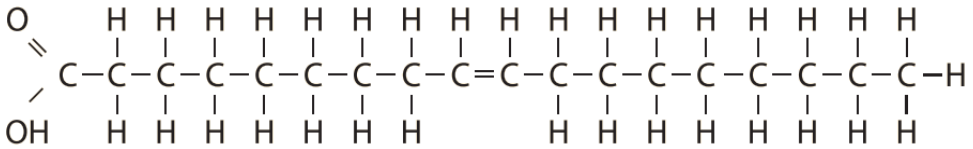

Figure 2-2 Monounsaturated fatty acids: oleic acid

If a fatty acid has more than one double bond within the fatty acid chain, it will be identified as polyunsaturated fatty acids. There are polyunsaturated fatty acids in the palm oil which are linoleic acid, $C_{18:2}$ shown in Figure 2-3 and linolenic acid, $C_{18:3}$ in Figure 2-4. Linoleic acid has two carbon-carbon double bonds in it while linolenic acid has three carbon-carbon double bonds in the chemical structure.

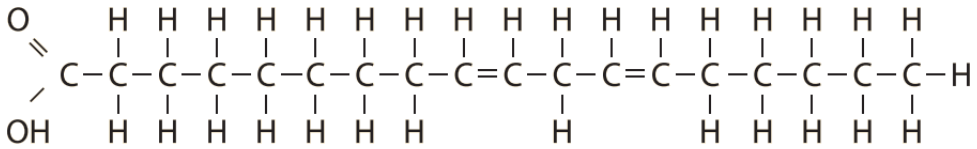

Figure 2-3 Polyunsaturated fatty acids: linoleic acid

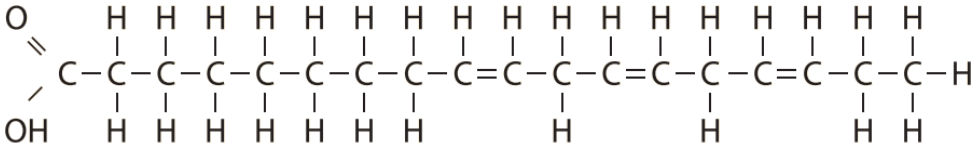

Figure 2-4 Polyunsaturated fatty acids: linolenic acid

2.2 Palm Oil

Palm oil is a type of consumable plant oil and derived from the flesh of fruit of oil palm *(Elaeis guineensis).* Due to the high content of beta-carotene, palm oil is bright

orange-red in colour. Palm oil is in semi-solid form at room temperature and has high resistance to oxidation and heat at high temperature for a long duration of time. Palm oil has replaced canola oil and soybean oil nowadays after putting performance efficiency and economical issue into the consideration.

Palm oil contains both saturated and unsaturated fatty acids in which unsaturated fatty acids will be further categorized into monounsaturated and polyunsaturated fatty acids. These saturated fatty acids include 39.389% palmitic, 3.915% stearic, 0.798% myristic, 0.243 lauric and 0.2% arachidic while unsaturated fatty acids include 43.829% oleic, 11.321% linoleic, 0.168% linolenic, 0.076% eicosenoic and 0.061% palmitoleic. (Duangmanee & Yoosuk,2012)

Palm oil is made up of triglyceride molecule that consists of carbon, hydrogen and oxygen atoms. Figure 2-5 shows the triglyceride structure that appears like an 'E' with glycerol and three long horizontal fatty acids attached to it.

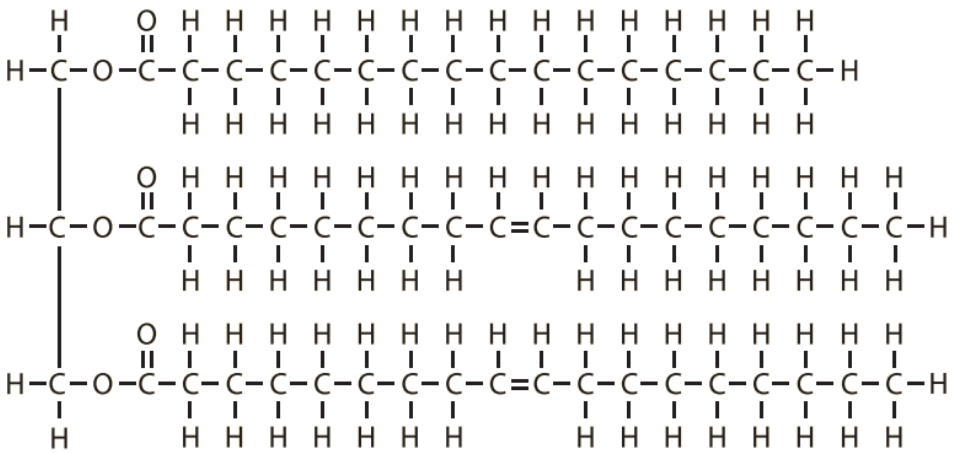

Figure 2-5 A typical palm oil triglyceride structure

2.3 Jatropha Oil

Jatropha curcas is a flowering plant under the Euphorbiaceae family. It is first found in American tropics such as Central America and Mexico and being cultivated in tropical regions around the world. (Janick, Jules; Robert E. Paull, 2008). This type of

plan can grow up to 6m in height and capable to live in extreme condition such as deserts due to its high resistance towards aridity. With the ability to live in extreme condition, Jatropha cursas do not face any problem in the cultivation process. However, most Jatropha plants are toxic and thus not suitable for human consumption as it can lead to diarrhea and strong vomiting.

Jatropha oil can be obtained from the crushing of the seed of Jatropha curcas. The seed from Jatropha curcas are readily to be used once it reaches its maturity when the capsule changes it colour from green to yellow. There are approximately 20% saturated fatty acids and 80% unsaturated fatty acids in the seeds which have great potential to be converted into Jatropha oil. (Nahar, K. and Ozores-Hampton, M.,2011) Jatropha oil produced can be used in many fields such as source of energy or fuel in term of biodiesel as it cannot be used for other purpose prior to detoxification process. Nowadays, Jatropha oil is being commonly used as an alternative for diesel as a renewable source as well as fuel for aviation purposes.

In the fatty acid composition of Jatropha oil, the saturated fatty acids include 14.2% palmitic, 7.0% stearic, 0.2% arachidic, 0.1% margaric and 0.1% myristic while unsaturated fatty acids include 44.7% oleic, 32.8% linoleic, 0.2% linolenic, and 0.7% palmitoleic. This gives a total of 21.6% of saturated fatty acids and 78.4% of unsaturated fatty acids present in Jatropha oil. (E.Akbar et al, 2009)

2.4 Rubber Seed Oil

Rubber tree, technically known as Hevea brasiliensis is a type of plant under the Euphorbiaceae, similar to Jatropha curcas. Rubber tree plantation had enhanced the economy due to high demand of milky latex extracted from rubber tree which can be used in the production of natural rubber. Applications of rubber tree are normally focused on the production of latex as the rubber woods and seeds are often neglected.

Rubber seed content great amount of fatty acids which include 20% of saturated fatty acids and 80% of unsaturated fatty acids. (Aigbodion, A. I., Pillai, C.S.T., 2000). Hence, extraction of rubber seed oil had been done previously in order extract the rubber seed oil for further industrial application. Studies had shown on the capability

of rubber seed oil to be used as fuel in the compression engines. (Ramadhas, A.S.; Jayaraj,S.; Muraleedharan,C, 2005). Furthermore, researches had also proved on its functionality to be used in the production of biodiesel. In which it has been found to be beneficial in production of biodiesel and compression engines fuels. (Ikwuagwu O. E.; Ononogbo, I. C.; Njoku, O. U, 2000).

Rubber seed oil contain significant number of fatty acids which include saturated fatty acids such as 7.9% of palmitic and 9.0% of stearic acid as well as unsaturated fatty acids include 28.9% of oleic, 40.5% lenoleic and 13.5% leolenic. This has totaled up 16.9% of saturated fatty acids and 82.9% unsaturated fatty acids. (Salimon,J. Ishak, A.A., 2012)

2.5 Fatty Acid Compositions of Palm Oil, Jatropha Oil and Rubber seed Oil

Table 2-1 Fatty acid composition of palm oil, Jatropha oil and rubber seed oil

Fatty Acid Composition	Palm Oil[a]	Jatropha Oil[b]	Rubberseed Oil[c]
Saturated			
Palmitic	39.389	7.9	14.2
Stearic	3.915	9.0	7.0
Myristic	0.798	-	0.1
Lauric	0.243	-	-
Arachidic	0.2	-	0.2
Margaric	-	-	0.1
Unsaturated			
Oleic	43.829	28.9	44.7
Linoleic	11.321	40.5	32.8
Linolenic	0.168	13.5	0.2
Eicosenoic	0.076	-	-
Palmitoleic	0.061	-	0.7
Total Saturated	**44.545**	**16.9**	**21.6**
Total Unsaturated	**55.455**	**82.9**	**78.4**

[a]: Reported in (Duangmanee & Yoosuk,2012)

[b]: Reported in (E.Akbar et al, 2009)

[c]: Reported in (Salimon,J. Ishak, A.A., 2012)

Comparison done on the fatty acids of palm oil, Jatropha oil and rubber seed oil in Table 2-1 shows greater percentage of unsaturated fatty acids present in Jatropha oil and rubber seed oil as compared to the one in palm oil. This explains on greater IV up to 105 gI_2/100g of oil for Jatropha oil and highest %OO produced which is 4.5 with 20wt% catalyst for 9 hours of reaction time (V.V.Goud et al,2007). Rubber seed oil with IV of 155.56gI_2/100g oil can produce up 3.8 %OO with its operating temperature set at 60^oC as reported in (Okieimen.F.E et al, 2001). These %OO are comparably higher as compared to palm oil which can reach up to %OO of 3.04 only with IV of 55gI_2/100g oil as reported in (Duangmanee& Yoosuk,2012).

2.6 Principle of Iodine Value

Iodine Value or more commonly known as IV is usually being used for the determination of unsaturation in particular oils. This unsaturation mainly refers to amount of double bonds present in oils itself. Greater amount of double bonds in particular oil will result in higher amount of IV. Besides giving information on the amount of double bonds present, IV do able to give information on the reactivity, stability and susceptibility to oxidation and rancidification of the oil.

Wijs solution which is the solutions of iodine monobromide and iodine monochloride in glacial acetic acid will be added to the fatty acid as shown in Figure 2.6.(AOAC Official Method of Analysis, 1984)

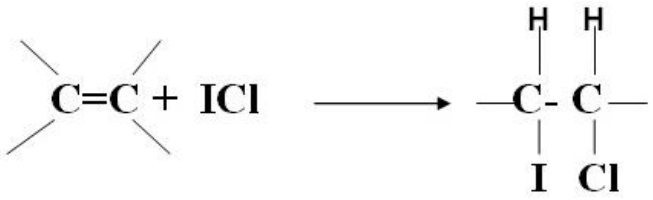

Figure 2-6 Double bond in fatty acid is reacted with iodine monochloride to produce di-halogenated single bond

The unreacted iodine monochloride or iodine monobromide will be reacted with potassium iodide to produce iodine as shown in Figure 2.7. The iodine will be titrated with $Na_2S_2O_3$ as shown in Figure 2.8 and starch will be added when it is near the end point to give purple colour product so that end point can be observed.

$$\mathbf{ICI + KI \longrightarrow KCI + I_2}$$

Figure 2-7 Reaction of iodine monochloride with potassium to produce iodine

$$\mathbf{I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_2O_4}$$

Figure 2-8 Reaction of iodine being titrated with sodium thiosulfate

2.7 Principles of Epoxidation Process

Epoxidation is a chemical reaction of converting carbon-carbon double bond to epoxide functional group consists of a three-member ring with two carbon atoms and one oxygen atom as shown in Figure 2-9. It has become a very important process nowadays as the epoxides obtained from the oil can be used as high temperature lubricants or raw materials for chemicals such as alcohols or glycols. Epoxides with higher oxirane values and lower iodine values are considered to be of better quality (Carlson and Chang, 1985; Hang and Yang, 1999).

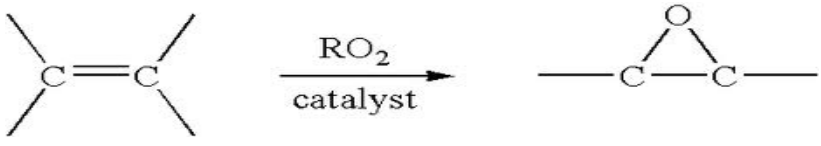

Figure 2-9 Epoxidized oil with carbon-carbon double bond being converted into oxirane ring

There are four technologies used to produce epoxides nowadays from olefinic-type of molecules. The selection of methods varies from case to case depending on the nature of the reactants and catalysts used for epoxidation. The four technologies are as follows:

i. Epoxidation with Percarboxylic Acids (Guenter et al., 2003)
    a. Widely used in industries
    b. Catalysed by acids or enzymes (Warmel and Klaas,1995; Rios et al., 2005)

ii. Epoxidation with Organic and Inorganic Peroxides
    a. Catalysed by transition metal catalysts (Sharpless et al., 1983)

iii. Epoxidation with Halohydrins
    a. Using hypohalous acids (HOX) and their salts as reagents (Guenter et al.,2003)

iv. Epoxidation with Molecular Oxygen (Guenter Et Al., 2003)
    a. Catalysed by silver

Epoxidation with halohydrins is an environmental unfriendly process. Epoxidation with molecular oxygen is the cheapest and greenest process among all but it is only restricted to simple molecules or substrates such as ethylene and butadiene owing to very low yields in the case of other alkenes (Dinda et al, 2008). Furthermore, oil will degrade to low molecular weight volatile compounds such as ketones, and short chain dicarboxylic acids which will make the process less selective for epoxides and thus reduce the efficiency and yield.

From the four technologies listed above, epoxidation with percarboxylic acids and epoxidation with organic and inorganic peroxides are suitable for clean and efficient epoxidation for vegetable oils. They can be rendered cleaner by using heterogeneous catalysts instead of traditional homogeneous catalysts (Campanella et al., 2004).

Epoxidation process is shown in Figure 2-10 for both first and second reaction respectively. Reversible reaction between carboxylic acids and hydrogen peroxide form peracid which subsequently reacts with vegetable oils at the 2nd reaction. Addition of hydrogen peroxide is essential in the epoxidation process to produce epoxide groups. Application of catalysts such as liquid inorganic acids or acidic ion exchange resins helps in producing higher yield of epoxides group.

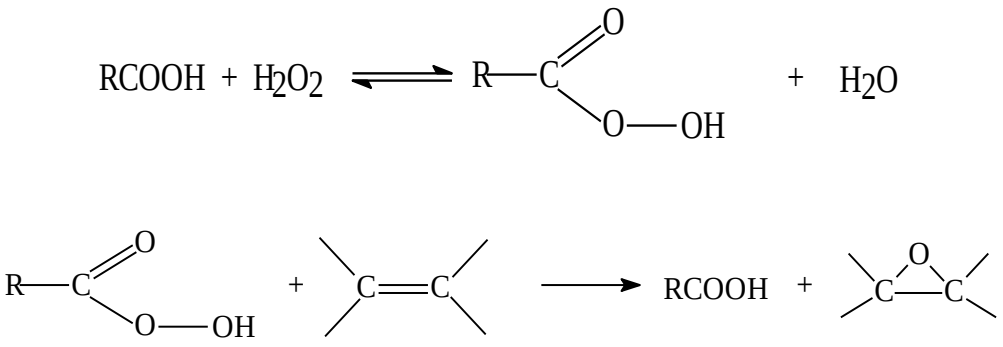

Figure 2-10 Main reactions in epoxidation process

Generation of peracids can be accelerated by strong mineral acids such as sulfuric acids. Decomposition of generated peracids might occur if the rate of epoxidation is slower than rate of generation of peracids. This will result in a lower yield of epoxidation process based on the consumed hydrogen peroxide. Different reaction parameters should be considered to ensure higher rate of epoxidation process to ensure higher yield of oxirane ring. (Milchert.E & Smagowicz.A, 2009)

However, consideration and attention should be put in the side reaction of the epoxidation process as shown in Figure 2.11. The decomposition of epoxy group will happen due to hydrolysis or acylation as shown in the 1st reaction (Campanella A, Baltanas M.A.,2006). Other possible side reactions of epoxides are shown in 2nd reaction which involves the reaction of epoxides with acids (Gamage P.K.et al, 2009). In other word, these reactions will lead to the degradation of oxirane rings and it will produce other side products which are undesirable. Minimization of these side products is essential to produce higher yield of epoxides or oxirane oxygen.

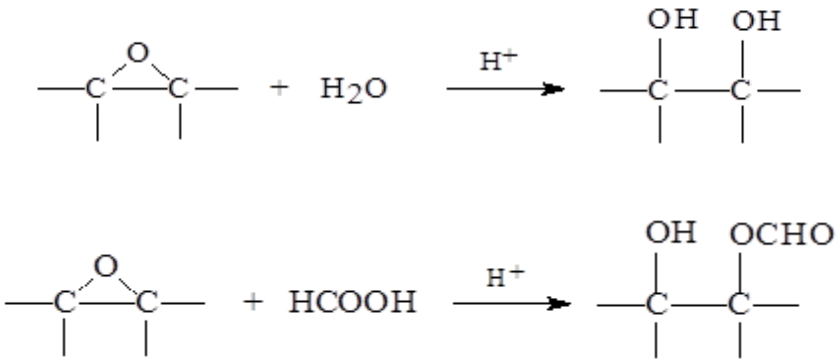

Figure 2-11 Side reactions in epoxidation process

2.8 Comparison of IV and %OO of Oil Undergo Epoxidation Process

Comparison between different types of oil that undergo epoxidation process was done. The IV of the particular oil and its optimum %OO achievable after optimization study were tabulated out as shown in Table 2-2. It can be concluded that the IV will affect directly the optimum %OO that can be achieved by particular oil itself. Higher amount of IV of the oil will lead to higher amount of %OO that was achieved after the epoxidation process.

Table 2-2 Comparison of IV and %OO of oil undergo epoxidation process

Literature Review	Type of Oil	Catalyst	Iodine Value (g I_2/100g oil)	Optimum %OO
F.E.Okieman et al (2010)	Rubber seed Oil	Sulfuric Acid	155.6	3.80*
Meshram et al (2011)	Wild Safflower Oil	Amberlite IR 122	155	7.87
T.I.Conney et al (2011)	Hemp Oil	Amberlite IR-120	133	6.81
Sinadinovic Fiser et al (2001)	Soybean Oil	Amberlite IR 120	130	5.99
R.Mungroo et al (2008)	Canola Oil	Amberlite IR-120H	112	5.94
S.Dinda et al (2008)	Cottonseed Oil	Sulfuric Acid	106	4.88
V.V.Goud et al (2007)	Jatropha Oil	Amberlite IR-120	105	4.50
S.Dinda et al (2010)	Cottonseed Oil	Amberlite IR-120	105	4.04
V.V.Goud et al (2006)	Mahua Oil	Amberlite IR-120	88	5.78
V.V.Goud et al (2007)	Mahua Oil	Sulfuric Acid	88	3.80
Sinadinovic Fiser et al (2012)	Castor Oil	Amberlite IR 120	81.5	3.83
Duangmanee & Yoosuk(2012)	Palm Oil	Sulfuric Acid	55	3.04

* : Study with no optimization study done

2.9 Comparison of Operating Parameters for Optimization of Epoxides

Table 2-3 Summary of information gathered from literature review on the optimum condition for epoxidation process

Literature Review	**Type of Oil**	**Stirring Speed (rpm)**	**C=C:Acetic Acid: H_2O_2 (moles)**	**Catalyst**	**Catalyst Concentration (%)**	**Temperature (^oC)**
V.V.Goud et al (2006)	Mahua Oil	1500	1:0.5:1.1	Sulfuric Acid	2.0	55-65
V.V Goud et al (2006)	Karanja Oil	1500	1:05:1.5	Amberlite IR-120	-	55-65
S.Dinda et al (2008)	Cottonseed Oil	1800	1:0.5:2	Sulfuric Acid	2.0	50-60
R.Mungroo et al (2008)	Canola Oil	-	1:0.5:1.5	Amberlite IR-120H	22	65
E.Milchert et al (2009)	Rapeseed Oil	500	1:1.12:9.5	-	-	60
A.Campanella and M.A.Baltanas (2005)	Vegetable Oil	1000	-	Sulfuric Acid	-	50-80

Literature Review	Type of Oil	Stirring Speed (rpm)	C=C:Acetic Acid: H_2O_2 (moles)	Catalyst	Catalyst Concentration (%)	Temperature (^oC)
Duangmanee & Yoosuk(2012)	Palm Oil	500	1:1:7	Sulfuric Acid	2	75
Meshram et al (2011)	Wild Safflower Oil	2000	1:0.5:1.5	Amberlite IR 122	20	60
Sinadinovic Fiser et al (2001)	Soybean Oil	1000	1:0.5:1.1	Amberlite IR 120	5	75
Sinadinovic Fiser et al (2012)	Castor Oil	1500	1:0.5:1.5	Amberlite IR 120	10	50
T.I.Conney et al (2011)	Hemp Oil	150	1:0.35-0.7:1	Amberlite IR-120	15	75
V.V.Goud et al (2007)	Mahua Oil	1500	1:0.5:1.1	Amberlite IR-120	16	70
S.Dinda et al (2010)	Cottonseed Oil	1800	1:0.5:1.5-2.0	Amberlite IR-120	15	75
F.E.Okieman et al (2010)	Rubberseed Oil	-	-	Sulfuric Acid	-	50-60

2.10 Application of Epoxides in Industries

The application of epoxides in the industrial field becomes very important, considering on the environmental effects of petroleum oil used widely nowadays. Vegetable oils are renewable raw materials with good lubricity, law volatility, high viscosity index and solvency for lubricants additives. They can be chemically or enzymatically treated to replace materials derived from petroleum. The main applications of epoxides are listed as belows:

- Act as raw materials for a variety of chemicals such as alcohols, glycols, alkanolamines, carbonyl compounds, olefinic compounds and polymers such as polyesters, polyurethanes, and epoxy resins. (V.V.Goud et al, 2008).
- Used directly as plasticisers which are compatible with polyvinyl chloride (PVC). (Milchert, E. & Smagowicz, A., 2009)
- Act as stabilizers for PVC resins to improve flexibility, elasticity, and toughness and high-temperature lubricants
- Impart the stability of polymer towards heat and UV radiation (V.V.Goud et al, 2008).
- Improve the properties of a large variety of polymers, as lubricants and detergents, in the production of ceramic coatings and paints and more recently in cosmetics and pharmaceuticals formulations. (Campanella A, Baltanas MA., 2005)
- Products obtained from the ring opening can be employed as low-temperature lubricants.
- Improve the elasticity of plastics and used in production of packing materials such as wrapping foils.
- Used for both formation of thermosetting composites and coatings obtained by ultraviolet (UV) –initiated cross-linking. (Milchert, E. & Smagowicz, A., 2009)

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1 Research Methodology/ Project Activities

All the project activities that are required for the project are summarized in Figure 3-1. The details of the methodology in the study of optimization of IV for the oil mixture will be explained in the next section.

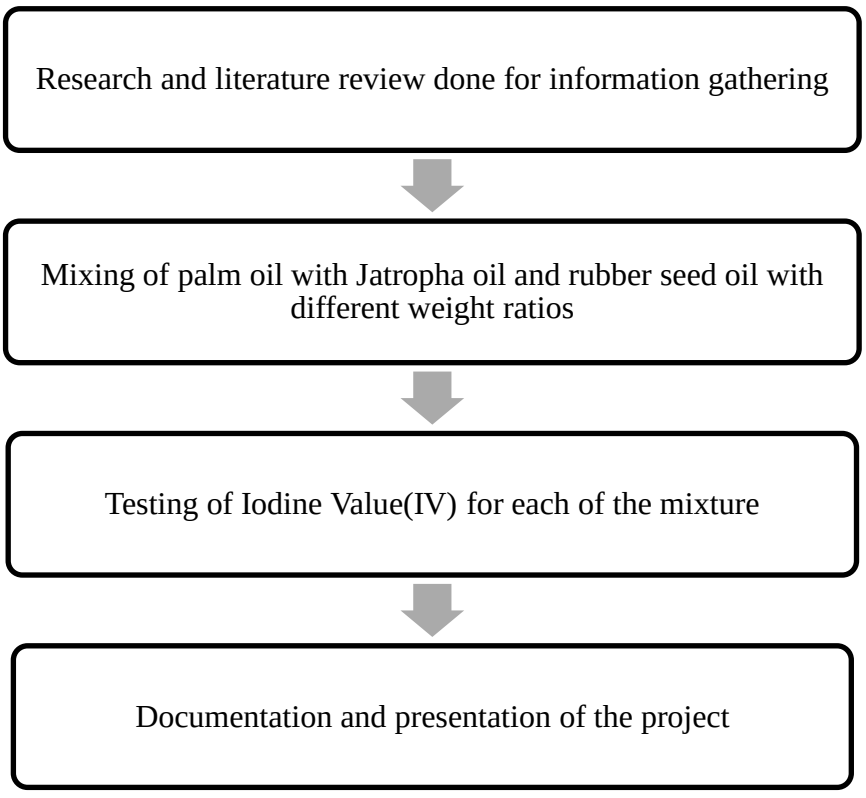

Figure 3-1 Project flow chart

The mixing between edible and non-edible oil at different weight ratio have been carried out. The combination and their respective weight ratios are identified at the beginning and tested on the IV. The calculation on the $\%OO_{TH}$ will be done after the mixture of oil undergo IV testing. Since the objective of the project is to increase the amount of IV in palm oil by mixing with other types of non-edible oils, palm oil will be remained in the combination while mixing. The combination between edible oil and non-edible oil can be arranged as follows:

a. Palm oil and Jatropha oil

b. Palm oil and rubber seed oil

c. Palm oil, Jatropha oil and rubber seed oil

Different weight ratios of oil are mixed to produce a new mixture of edible and non-edible oil. The weight ratios are pre-determined by using Design Expert Software specifically on Mixture. From the tabulation generated in the software, 8 runs are required for the mixture of palm oil and Jatropha oil as well as the mixture of palm oil and rubber seed oil. Furthermore, 14 runs are required for the mixture of palm oil, Jatropha oil and rubber seed oil. Hence, a total of 30 runs of mixing at different weight ratio have been conducted and each of the mixture's IV is tested. The theoretical percentage oxirane oxygen ($\%OO_{TH}$) has been calculated based on the IV value tested.

The IV of each mixture is tested with the titration of $Na_2S_2O_3$, following the AOAC Official Method of Analysis. The raw data obtained from the experiments are used as raw data in Design Expert Software. Design Expert Software will be able to identify the optimum weight ratio for all three types of combinations with the highest amount of IV and $\%OO_{TH}$.

Table 3-1 and Table 3-2 relatively show all 8 runs in each of the combination. The weight ratios are determined by the Design Expert Software itself and experimental testing should be conducted to identify the IV for each of weight ratio.

Table 3-1 Experimental runs for mixture of palm oil and Jatropha oil

Run	Weight Percentage of Palm Oil (%)	Weight Percentage of Jatropha Oil (%)	Iodine Value (g I_2/100g)	$\%OO_{TH}$
1	100	0		
2	50	50		
3	0	100		
4	75	25		
5	25	75		
6	0	100		
7	100	0		
8	50	50		

Table 3-2 Experimental runs for mixture of palm oil and rubber seed oil

Run	Weight Percentage of Palm Oil (%)	Weight Percentage of Rubber Seed Oil (%)	Iodine Value (g I_2/100g)	$\%OO_{TH}$
1	100	0		
2	50	50		
3	0	100		
4	75	25		
5	25	75		
6	0	100		
7	100	0		
8	50	50		

Table 3-3 shows the experimental runs identified for the combination of palm oil, jatropha oil and rubber seed oil mixture with different weight ratio respectively.

Table 3-3 Weight ratio of mixture between palm oil, Jatropha oil and rubber seed oil

Run	Weight Percentage of Palm Oil (%)	Weight Percentage of Jatropha Oil (%)	Weight Percentage of Rubber Seed Oil (%)	Iodine Value (g I_2/100g)	$\%OO_{TH}$
1	100.00	0.00	0.00		
2	50.00	50.00	0.00		
3	50.00	0.00	50.00		
4	0.00	100.00	0.00		
5	0.00	50.00	50.00		
6	0.00	0.00	100.00		
7	66.67	16.67	16.67		
8	16.67	66.67	16.67		
9	16.67	16.67	66.67		
10	33.33	33.33	33.33		
11	100.00	0.00	0.00		
12	0.00	0.00	100.00		
13	0.00	100.00	0.00		
14	50.00	50.00	0.00		

Several methods were well-established and recognized to be used to test the Iodine Value for a certain oil sample nowadays. In this project, the Iodine Value testing will be carried out by using a standard testing proposed in (AOAC Official Method of Analysis, 1984).The procedure of testing is summarized as follows:

1. 0.1g of oil sample is taken into a 250ml of glass stopper iodine flask.
2. Oil sample is dissolved in 20ml of chloroform and 25ml of Wijs solution.
3. It is shake well and place in dark for 30 minutes.

4. 20ml of 15% potassium iodide (KI) and 100ml of distilled water is added in the flask.
5. The content was titrated with 0.1N sodium thiosulfate anhydrous using starch as indicator until yellow iodide colour is disappeared as shown in Figure 3-2.

Figure 3-2 Apparatus set up for titration in Iodine Value Testing

6. Same procedure is done for blank solution with the exception that no oil is added in the sample.
7. Iodine Value can be calculated by the following formula:

$$\text{IV} = \frac{(\text{Blank Titration-Sample Titration}) \times \text{Normality of } Na_2S_2O_3 \times 12.69}{\text{Weight of Sample}}$$

Where Blank Titration = Volume of $Na_2S_2O_3$ titrated for blank;

Sample Titration = Volume of $Na_2S_2O_3$ titrated for sample;

Normality of $Na_2S_2O_3$ = 0.1N

3.2 Tools/Hardware

The experimental work for the project requires basic chemical equipment such as burette, pipette, beakers, measuring cylinders and conical flasks for IV testing. Aluminium foils are used to close the conical flasks while being stored in a dark cabinet for IV testing purposes. Other common equipment used in this experimental work includes volumetric flasks, weighing machine, spatula and glassware for sample and chemical preparation.

3.3 Key Milestones

The key milestone in this project mainly focuses in several sections in order to ensure the objective of the project can be achieved within the time period. The key milestones identified in this project are:

1. Literature review on plasticizer application
    - Literature review has to be done in order to identify the amount of %OO for plasticizers application.
    - The amount of %OO identified is used as a standard to be achieved in the optimization of IV studies.

2. Optimization study for Iodine Value of oil mixture
    - All three types of combination of oil mixture are carried out.
    - The optimum weight ratios for each types of combination is selected based on the standard identified earlier.
    - Literature review should be done to support on valid reason why the certain weight ratio of oil is being chosen.
    - The best weight ratio or combination is chosen and justified based on the literature review done.

3. Documentation and presentation of project
    - Documentation of the project including dissertation and technical paper will be prepared.

- Results and discussion made from the experimental data obtained will be reported.
- Further discussion made on the recommendation for the project future works.

Figure **3-3** shows complete Gantt chart for the final year project and certain important milestones identified along the duration.

3.4 Gantt Chart

NO	DETAIL / WEEK	1	2	3	4	5	6	7	Mid Semester Break	8	9	10	11	12	13	14
1	Study of IV for mixture of Palm oil and Jatropha oil		●													
2	Study of IV for mixture of Palm oil and Rubber seed oil						●									
3	Study of IV for mixture of Palm oil, Jatropha oil and Rubber seed oil										●					
4	Pre-Sedex															
5	Submission of Draft Report															
6	Submission of Softbound Dissertation													●		
7	Submission of Technical Paper													●		
8	Oral Presentation														●	
9	Submission of Final Project Dissertation															●

●Suggested Milestone

Figure 3-3 Gantt chart for final year project

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Data Gathering and Analysis for Optimization of Iodine Value

The study to optimize the iodine value (IV) for the mixture between edible oil and non-edible oil was carried out. Theoretically, the theoretical percentage oxirane oxygen (%OO_{TH}) for each type of oil can be calculated if the IV of the oil sample is known. In the project, the %OO_{TH} for each run is calculated using the formula below based on the IV obtained from experimental data.

$$OO_{TH=}\frac{\frac{IV_o}{2A_i}}{100+\left(\frac{IV_o}{2A_i}\right)A_o}\times A_o \times 100$$

Where, A_i (126.9) is the atomic weight of iodine;

A_0 (16.0) is the atomic weight of oxygen and;

IV_0 is the initial iodine value of the oil sample.

However, it is often observed that the percentage oxirane oxygen (%OO) for epoxidized vegetable oils is less than the %$OO_{TH.}$ This can be explained from the relative conversion of double bonds to epoxide group that do not reach 100% from the epoxidation process carried out. The experimental data are tabulated and it includes the IV for each type of mixture of oil as well as the %OO_{TH} that can be obtained from the respective mixture of oil. The raw data for each of the mixture are tabulated in the APPENDIX A.

For all the data analysis of three different combination of oil, one standard %OO is being chosen which act as a reference value. The application of epoxidized rubber seed oil (ERSO) as coupling agent and plasticizers in silica-filled natural rubber compounds was found to be effective at %OO of 5 (Reethamma,J et al , 2004). Taking relative conversion of 90% into consideration, the $\%OO_{TH}$ that is the most desirable would be 5.5 to 5.6 in order to obtain %OO of 5 in the epoxidation process.(Klaas, M.R. et al,1999)

4.1.1 Data Gathering for Mixture of Jatropha Oil and Palm Oil

Table 4-1 shows the data tabulation for the mixture of Jatropha oil and palm oil. It shows the IV for each mixture and it's respectively $\%OO_{TH}$. The data is used in Design Expert Software. In the software, the desired value of $\%OO_{TH}$ is set to be 5.5 and the result stimulated will show what weight ratio of Jatropha oil and palm oil will give the desired result.

Table 4-1 Data tabulation for mixture of Jatropha oil and palm oil

Run	Palm Oil (wt%)	Jatropha Oil (wt%)	Iodine Value	$\%OO_{TH}$
1	100	0	65.18	3.95
2	100	0	70.95	4.28
3	0	100	106.71	6.30
4	0	100	100.94	5.98
5	50	50	83.12	4.98
6	50	50	77.50	4.66
7	25	75	96.44	5.73
8	75	25	73.26	4.41

Figure 4-1 shows the graph of $\%OO_{TH}$ versus weight ratio for the mixture of Jatropha oil and palm oil. The main objective is to find the optimum weight ratio to give out the most desired $\%OO_{TH}$ which is 5.5 as identified earlier. In this case, analyzed data shows that with 27.85 wt% of palm oil and 72.15 wt% of Jatropha oil can produce $\%OO_{TH}$ up to 5.5 with desirability of 1.00.

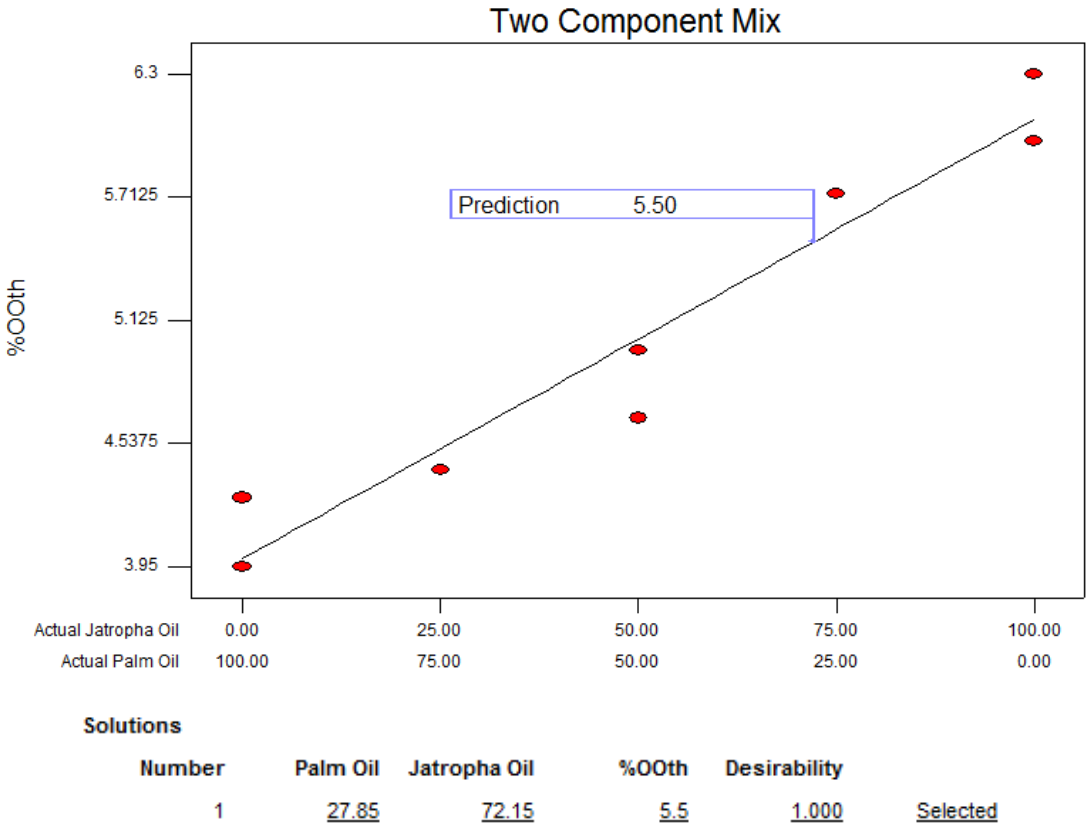

Figure 4-1 Graph of $\%OO_{TH}$ versus weight ratio for Jatropha oil and palm oil

4.1.2 Data Gathering for Mixture of Rubber Seed Oil and Palm Oil

Table 4-2 shows the data tabulation for mixture of rubber seed oil and palm oil. It shows the IV of each combination and the respective $\%OO_{TH}$. The data is analyzed to investigate at which combination of weight ratio will produce the desired result.

Table 4-2 Data tabulation for mixture of rubber seed oil and palm oil

Run	Palm Oil (wt%)	Rubber Seed Oil (wt%)	Iodine Value	$\%OO_{TH}$
1	100	0	65.18	3.95
2	100	0	70.95	4.28
3	0	100	138.96	8.05
4	0	100	125.17	7.31
5	50	50	90.56	5.40
6	50	50	95.18	5.66
7	25	75	127.53	7.44
8	75	25	89.41	5.34

Figure 4-2 shows the graph of $\%OO_{TH}$ versus weight ratio for the mixture of rubber seed oil and palm oil. From the data analysis, it is found out that a mixture with 38.21 wt% of rubber seed oil and 61.79 wt% of palm oil can achieve $\%OO_{TH}$ of 5.5 with desirability up to 1.00.

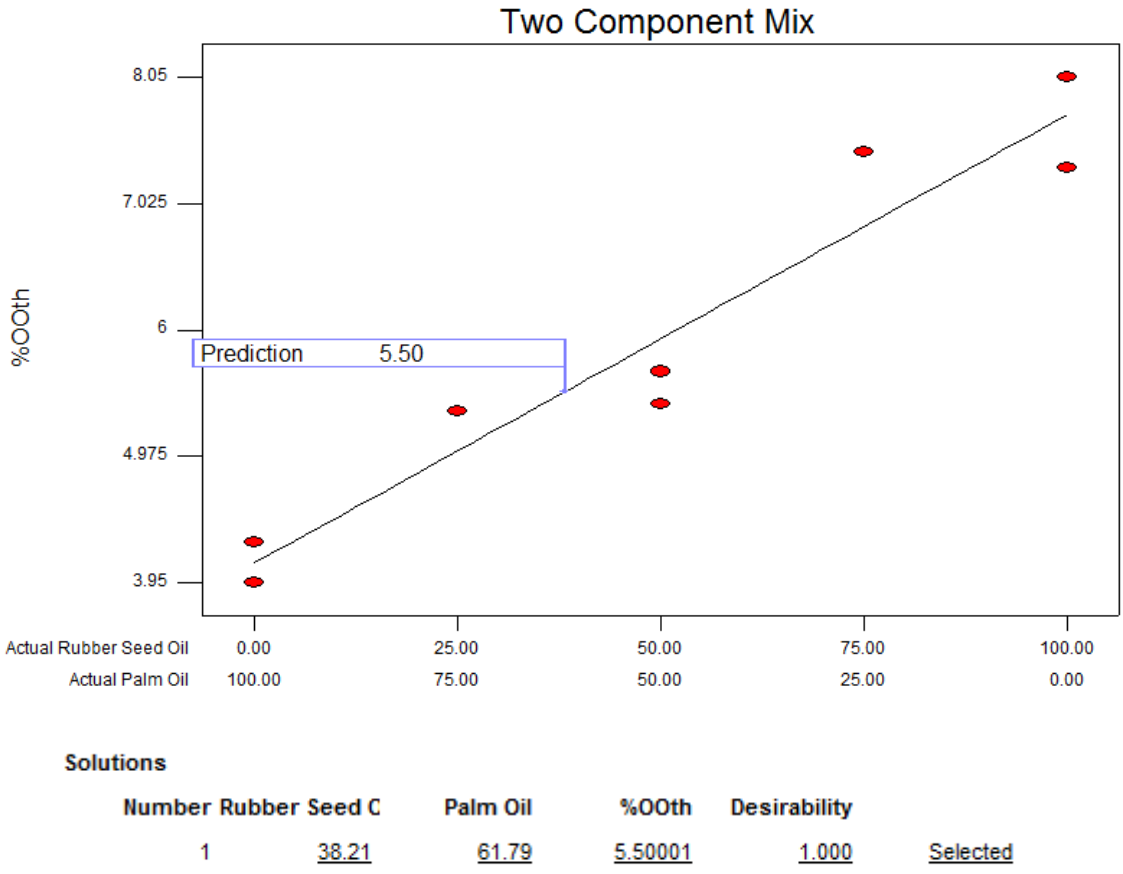

Figure 4-2 Graph of $\%OO_{TH}$ versus weight ratio for rubber seed oil and palm oil

4.1.3 Data Gathering for Mixture of Rubber Seed Oil, Jatropha Oil and Palm Oil

Table 4-3 shows the data tabulation for mixture of rubber seed oil, Jatropha oil and palm oil. It shows the IV of each combination and the respective $\%OO_{TH}$. Similar to previous data analysis, the experimental data will be used to investigate the optimum weight ratio to produce the desired $\%OO_{TH}$ of 5.5.

Table 4-3 Data tabulation for mixture of rubber seed oil, Jatropha Oil and palm oil

Run	Palm Oil (wt%)	Jatropha Oil (wt%)	Rubber Seed Oil (wt%)	Iodine Value	%OO_{TH}
1	100	0	0	65.18	3.95
2	100	0	0	70.95	4.28
3	0	100	0	106.71	6.30
4	0	100	0	100.94	5.98
5	0	0	100	138.96	8.05
6	0	0	100	125.17	7.31
7	50	50	0	83.12	4.98
8	50	0	50	90.56	5.40
9	0	50	50	124.02	7.25
10	50	50	0	77.50	4.66
11	66.67	16.67	16.67	84.79	5.07
12	16.67	66.67	16.67	99.79	5.92
13	16.67	16.67	66.67	113.58	6.68
14	33.33	33.33	33.33	110.17	6.49

Figure 4-3 shows seven possible results with the desired objective to obtain %OO_{TH} of 5.5. The combination that takes the average amount of all three types of oil is being selected and graphical presentation is displayed in Figure 4-4.

Solutions

Number	Rubber Seed C	Jatropha Oil	Palm	*OOth	Desirability	
1	20.24	32.55	47.21	5.49999	1.000	Selected
2	29.99	16.26	53.76	5.50001	1.000	
3	8.24	52.63	39.14	5.5	1.000	
4	9.83	49.96	40.21	5.49999	1.000	
5	35.40	7.21	57.39	5.50001	1.000	
6	0.48	65.60	33.92	5.49999	1.000	
7	4.34	59.15	36.52	5.50001	1.000	

7 Solutions found

Figure 4-3 Seven possible combinations to obtain %OO_{TH} of 5.5.

Figure 4-4 shows graph of $\%OO_{TH}$ versus weight ratio for the mixture of rubber seed oil, Jatropha oil and palm oil. From the data analysis, it is found out that a mixture with 20.24 wt% of rubber seed oil, 32.55 wt% of Jatropha oil and 47.21 wt% of palm oil can achieve $\%OO_{TH}$ of 5.5 with desirability up to 1.00.

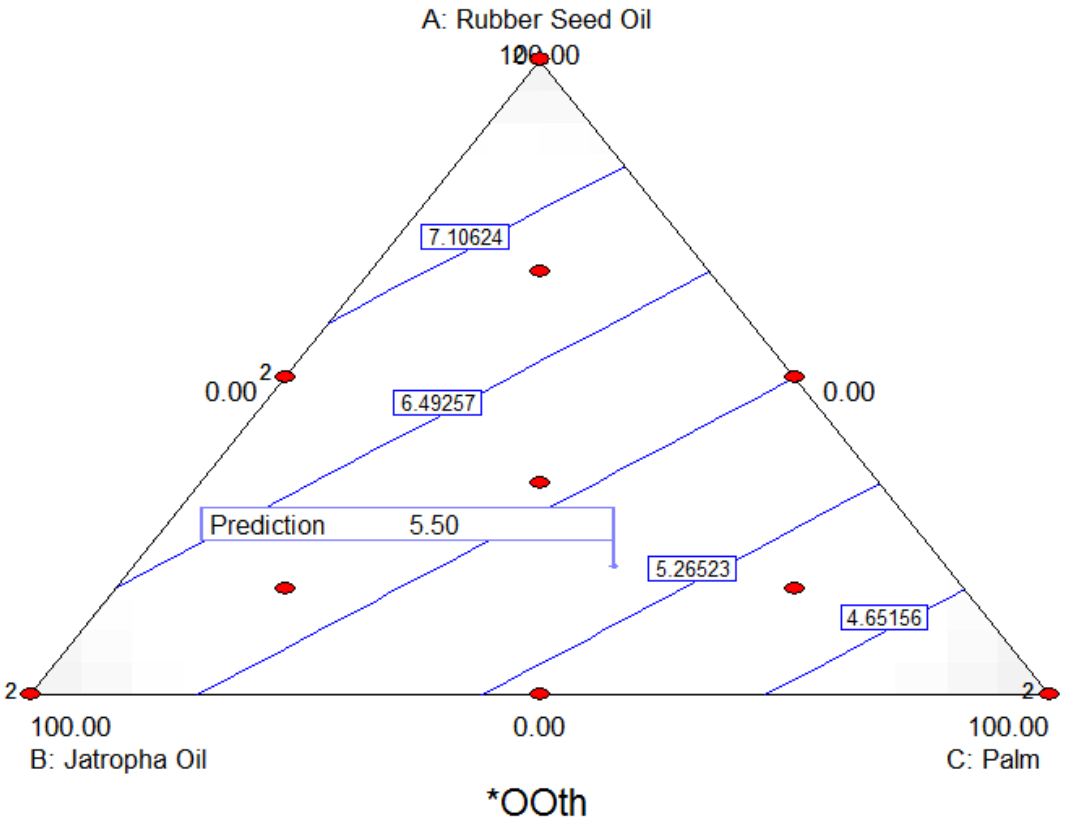

Figure 4-4 Graph of $\%OO_{TH}$ versus weight ratio for mixture of rubber seed oil, Jatropha oil and palm oil

4.1.4 Data Analysis for Optimization of Iodine Value

Summarizing from the data obtained, these data are further analyzed to investigate the most suitable combination of oil with its respective weight ratio to be used in the study of optimization of epoxidation process. The selection is supported with rigid factual arguments from literature review.

Table 4-4 summarizes all the data obtained from the optimization of IV studies for all three types of oil combination. These compositions are able to yield up to 5.5 of $\%OO_{TH}$ with desirability up to 1.00.

Table 4-4 Summary of data obtained for all optimization of IV studies

	Composition (wt%)		
	Rubber seed oil	Jatropha oil	Palm oil
Mixture of Jatropha oil and palm oil	-	72.15	27.85
Mixture of rubber seed oil and palm oil	38.21	-	61.79
Mixture of rubber seed oil, Jatropha oil and palm oil	20.24	32.55	47.21

For all three type of oil combination, data analysis from Design Expert Software shows a **linear relationship** for the mixing. Mixing between non-edible oils with higher IV and edible oil with lower IV will result in a mixture of oil having an averaged IV, relatively based on the weight ratio of each type of oil in the particular combination. From Table 4-4, higher composition of Jatropha oil is required in the oil mixture between Jatropha oil and palm oil as compared to the oil mixture between rubber seed oil and palm oil that required less amount of rubber seed oil. This can be explained with the fact that rubber seed oil contains higher amount of unsaturated fatty acids such as linoleic and linolenic acids with more than one double bond in the structures. (Salimon,J. Ishak, A.A., 2012) Hence, less amount of rubber seed oil is required in the mixture of palm oil and rubber seed oil to achieve $\%OO_{TH}$ of 5.5. Jatropha oil with fewer amounts of unsaturated fatty acids will take up higher portion in the mixture to achieve the same standard. (E.Akbar et al, 2009) For the combination with three types of oils, the amount of palm oil required is the highest since both Jatropha oil and rubber seed oil with higher amount of unsaturated fatty acids are present in the oil mixture. From the linear relationship pattern shown, it can be concluded that the IV of the oil mixture can be increased by increasing the amount

of non-edible oils with higher IV or vice versa.

Studies had proven that the epoxidized oil with high value of %OO is categorized as high quality epoxidized oil (Carlson and Chang, 1985; Hang and Yang, 1999). Hence, apart from having high %OO in the epoxidation process, the selected combination of oil mixture with the respective weight ratio has to be able to meet the industrial standard in any applications such as coatings or plasticizers.Research carried out on the mechanical performance of EPO in coatings on metal surface showed that EPO-epoxy coating has good adhesive strength and impact strength. The EPO modified epoxy coatings only use 30 wt% of EPO together with the commercialized resins to show relatively good mechanical properties as compared with the application of resin in epoxy coatings(R.M. Taharim et al, 2012). Studies carried out on the effect of EPO on the properties and performances of epoxy resin showed that the EPO modified epoxy resin possesses good thermal stability and improve on strain and toughness with 20 wt% of EPO modified epoxy resin (Sarwono, 2009). Meanwhile, studies on ERSO as coupling agent and plasticizer showed that its application works the best with combination of ERSO and the commercialized coupling agents such as bis(triethoxy silyl propyl)tetrasulfane (TESPT). This combination shows an improvement in properties such as hardness, tear strength and compression set. However, it leads to a higher heat build-up or thermal instability due to low level of crosslinking of matrix (Reethamma,J et al , 2004).

These studies proved that it is possible with the mixing of EPO with ERSO, the thermal instability can be improved and the overall mechanical performance can be enhanced. Hence, 25wt% of palm oil and 75wt% of rubber seed oil is chosen as the optimum weight ratio of oil mixture after considering both experimental results as well as literature review. This oil combination with the weight ratio is expected to yield $\%OO_{TH}$ of 7.44. Mixing of these two edible and non-edible oils together is expected to increase low %OO of EPO and improve thermal stability of ERSO. Optimization study done on other type of combinations do suggest on the possibility that rubber seed oil can be replaced by Jatropha oil to yield a high $\%OO_{TH}$. Combination of three types of oil can also be carried out for the sake of same purpose.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The epoxidation of palm oil is concluded to be a good approach for the replacement of petroleum oil. This is a much interesting finding for countries such as Malaysia, Thailand, and Indonesia that are rich in the raw material which is the palm oil in this technology. However, the optimum yield of epoxides from EPO is lower compared to other types of oil such as EJO or ERSO. The optimization of the IV for palm oil in this project is done by mixing palm oil with Jatropha oil and rubberseed oil which have relatively higher amount of IV that result in higher $\%OO_{TH}$. It has been identified that the weight ratio of the oil mixture and the resulting IV is in a linear relationship. Mixing non-edible oils such as rubber seed oil and Jatropha oil with edible oil such as palm oil would result in an oil mixture with averaged value of IV. Oil mixture with higher amount of IV can be produced by having greater portion of non-edible oil in the mixture and vice versa.

The optimum weight ratio in order to obtain desired amount of IV and $\%OO_{TH}$ has been identified based on previous study which is 25 wt% of palm oil and 75 wt% of rubber seed oil. This weight ratio can produce $\%OO_{TH}$ up 7.44 which is much higher than the standard required in plasticizers application. The weight ratio selected can increase the IV of palm oil and is expected to possess a better thermal stability as compared to ERSO in plasticizers application.

5.2 Recommendations

There are a few recommendations for this project. Due to the time and financial constraints, certain scopes have been identified first in order to meet the objectives of the project. One of the recommendations that are suggested is to study further details on the optimum operating parameters required by the oil mixture for epoxidation process. These parameters include temperature, reaction time, mole ratio of double bonds and acetic acid, mole ratio of double bonds and hydrogen peroxide as well as catalyst loading. The levels in each parameter can be determined by using Design Expert Software, specifically in Response Surface Methodology. With the application of the software, the optimum operating parameters can be identified and it should be further clarified with actual experimental work. The optimum operating parameters that are identified can be used to yield higher amount of %OO as compared to the palm oil itself.

With the study of epoxidation that can be done, further study on the effectiveness of the epoxides produced from the oil mixture in the industrial application can be done. This includes mechanical performance and thermal stability in coatings or plasticizers. Previous study has shown that ERSO possess a greater thermal instability in plasticizers application while EPO shows a better thermal stability in coatings on metal surface. Comparison can be made with the previous mechanical and thermal stability performance study to investigate the significance of the mixing process.

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APPENDIX A

Table A- 1 Raw data for optimization study for mixture of Jatropha oil and palm oil

Experiment 1						
Mixing of Jatropha Oil and Palm Oil						
Run	Palm oil (wt%)	Jatropha Oil (wt%)	Weight (g)	Volume of sodium thiosulfate (ml)	Iodine Value	$\%OO_{TH}$
1	100	0	0.11	37.3	65.18	3.95
2	100	0	0.11	36.8	70.95	4.28
3	0	100	0.11	33.7	106.71	6.30
4	0	100	0.11	34.2	100.94	5.98
5	50	50	0.10	36.4	83.12	4.98
6	50	50	0.14	34.4	77.50	4.66
7	25	75	0.10	35.6	96.44	5.73
8	75	25	0.11	36.5	73.26	4.41

Table A- 2 Raw data for optimization study for mixture of rubber seed oil and palm oil

Experiment 2						
Mixing of Rubber Seed Oil and Palm Oil						
Run	Palm oil (wt%)	Rubber Seed Oil (wt%)	Weight (g)	Volume of sodium thiosulfate (ml)	Iodine Value	$\%OO_{TH}$
1	100	0	0.11	37.3	65.18	3.95
2	100	0	0.11	36.8	70.95	4.28
3	0	100	0.10	32.0	138.96	8.05
4	0	100	0.11	32.1	125.17	7.31
5	50	50	0.11	35.1	90.56	5.40
6	50	50	0.11	34.7	95.18	5.66
7	25	75	0.10	32.9	127.53	7.44
8	75	25	0.11	35.2	89.41	5.34

Table A- 3 Raw data for optimization study for mixture of rubber seed oil, Jatropha oil and palm oil

Experiment 3							
Mixing of Rubber Seed Oil, Jatropha Oil and Palm Oil							
Run	Palm oil (wt%)	Jatropha Oil (wt%)	Rubber Seed Oil (wt%)	Weight (g)	Volume of sodium thiosulfate(ml)	Iodine Value	$\%OO_{TH}$
1	100	0	0	0.11	37.3	65.18	3.95
2	100	0	0	0.11	36.8	70.95	4.28
3	0	100	0	0.11	33.7	106.71	6.30
4	0	100	0	0.11	34.2	100.94	5.98
5	0	0	100	0.10	32.0	138.96	8.05
6	0	0	100	0.11	32.1	125.17	7.31
7	50	50	0	0.10	36.4	83.12	4.98
8	50	0	50	0.11	35.1	90.56	5.40
9	0	50	50	0.11	32.2	124.02	7.25
10	50	50	0	0.14	34.4	77.50	4.66
11	66.67	16.67	16.67	0.11	35.6	84.79	5.07
12	16.67	66.67	16.67	0.11	34.3	99.79	5.92
13	16.67	16.67	66.67	0.10	34.0	113.58	6.68
14	33.33	33.33	33.33	0.11	33.4	110.17	6.49