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THE 5TH LUSTRUM

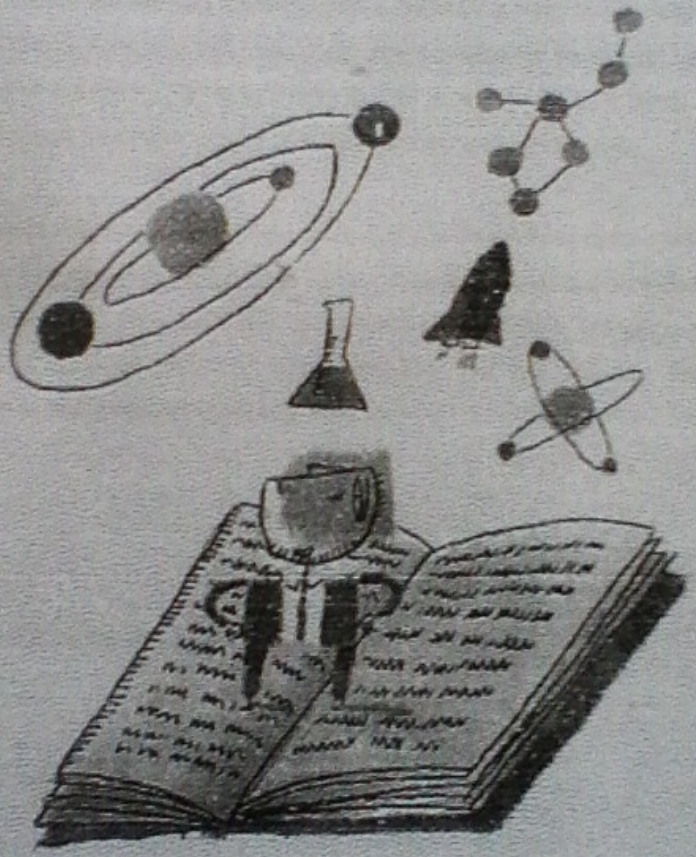
FACULTY OF MATHEMATICS AND NATURAL SCIENCES

AIRLANGGA UNIVERSITY

SURABAYA, August 6-7, 2007

UNAIR RuG KNAW UTM

international conference on Basic and Applied Sciences



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FOREWORD BY CHAIRPERSON

Basic science plays an important role in the development of advance industries, for example biotechnology, material science, informatics and nano technology. Based on these facts, faculty of Mathematics and Natural Sciences, Airlangga University in collaboration with Groningen University, The Royal Netherlands Academic of Art and Science, and Universiti Teknology Malaysia is organizing this conference and workshop. The conference is held on 6-7 August 2007 and will being attended by more than 150 participants. The workshop will be held on 7-8 August 2007 at Department of Chemistry, Airlangga University and more than 30 participants have registered. These participants come from several universities in Indonesia and Malaysia.

Finally, I would like to express our deep appreciation to the following guest speakers Prof. Bauke W. Dijkstra (Groningen University), Prof. Kazuo Sakka (Mie University), Prof Marsin Sanagi, Prof. Belrom Sanugi, and Prof. Noor Aini Abd. Rashid from Universiti Teknoiogy Malaysia, Dr. Ni Nyoman Tripuspaningsih, M.Si, Prof. Agoes Soegianto, Prof Win Darmanto, Prof. Suhariningsih and Dr. Eridani from Airlangga University.

Wassalamualaikum Wr. Wb

Dr.rer.nat. Ganden Supriyanto, M.Sc

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ABSTRACT OF INVITED LECTURES

OPTIMIZATION OF *Jatropha curcas* L. METHYL ESTER EPOXIDATION WITH AMBERLITE IR-120 CATALYST

Purwantiningsih Sugita, Ahmad Sjachriza, Budi Arifin dan Joko Soeharto

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ABSTRACT

The commonly utilized type of plasticizer is phtalate which is petrochemical-based, but in a periode of time may harm the environment. Therefore, it is important to find a substitute for phtalate that is not petrochemical based, one of which is vegetable oil. Shah (2003) reported that seed of *Jatropha curcas* oil contains highly about 40-60% (w/w) and it has a similarity of physical and chemical properties with *soybean* oil. For that reason, the *jatroph's* methyl ester (JME) is potentially developed elementary upon as a plasticizer. This research is consisted of by two stages (1) charactheristic analysis of JME and epoxide's (JMEE), and (2) synthesis and optimization of epoxidation reaction of JME use amberlite IR-120 catalyst. Optimization was carried out by three variations, reactions time and temperature were 12, 24, 36 hours and 50, 60, 70 °C, respectively and 1.0, 2.0, 3.0% amberlite IR-120 catalyst concentrations. The result showed that an iodine number in the epoxide (JMEE) was 5-6 times smaller than the JME, whereas oxyrane number was enhanced 84 times from initial condition. Optimum condition of epoxidation reaction were obtained at 70°C, 12 hours, and 3.0% catalyst concentration of amberlite IR-120. This condition showed that oxyrane number of research and *Modde 5.0* were 3.95% and 3.61%, respectively.

Keywords: JME, epoxidation and amberlite IR-120

INTRODUCTION

Phtalate is one of common plasticizer that is added into poly(vinyl chloride) (PVC) to make it flexible and imperishable. Phtalate comes from petroleum. The commonly used phtalates are dibutyl phtalate (DBP), dimethoxyethyl phtalate (DMEP), di-(2-ethylhexyl) phtalate (DEHP), dioctyl phtalate (DOP), and diisononyl phtalate (DIHP). DEHP is used in toys and medical tools, whereas DINP is used in toys (Syah 2003). Plasticizer from phtalate group can move into the surface of the plastic, then loose into environment. According to *EPA's Toxic Release Inventory*, phtalate compounds are toxic. Moreover, since 2006, Uni Europe has issued prohibition for using of phtalate ester as plasticizer (Tullo 2005). Therefore, environment-friendly substitute plasti-cizer is needed.

Plasticizer from vegetable oil has been developed, such as plasticizer from sunflower oil, rice oil, menhaden oil, oleic acid, and soybean oil (Chou & Lee 1997, Supriyadi 1992, and Hazimah *et al.* 2000). Plasticizer from vegetable oil that has dominated world market (43%) is epoxide of soybean oil (ESO). This plasticizer has superiority because it is biodegradable and renewable. Beside epoxide form vegetable oil, platicizer from vegetable oil ester, such as palm oil methyl ester has been done by Gan *et al.* (1992) and Wibowo *et al.* (2003).

Vegetable oil-based plasticizer or its methyl ester form can be synthetized by epoxidation. Vegetable oil epoxide can be done using *in situ* peroxyacetic acid, that is the product of reaction between acetic acid and hidrogen peroxide (Campanela dan Baltzanas 2005). In Indonesia, vegetable oil-based plasticizer has not ex-panded and the material are still imported, whereas in Europe and Japan, the using of this plasticizer has entered the application stage. This is ironic because Indonesia has a lot of vegetable resources, such as palm, pinnate, barn, rubber, and *jatropha curcas*.

Syah (2003) reported that *jatropha curcas* seed contains 40-60% (w/w) oil. The chemical and physical characteristic of *jatropha curcas* oil resemble soybean oil's. Gubitz *et al.* (1998) in Manurung (2005) reported that unsaturated fatty acids dominate this oil, especially oleic acid (34.3-45.8)% and linoleic acid (29.0-44.2)%. So in this research, the potent of JME as plasticizer will be investigated. This paper shows optimum condition for JME epoxidation with three variables, namely reaction time and temperature, and concentration of catalyst (amberlite IR-120). The parameters that was observed are iodine and oxyrane number. Optimum condition was obtained from respon surface method (RSM) in Modde 5.

METHOD

Synthesis and Optimization of JME Epoxidation (Chou & Lee 1997; Gan *et al.* 1992; Rangrajan & Harey 1995).

First, 0.80 ml glacial acetic acid, 2.90 ml toluene, and 1.0-3.0% amberlite IR-120 (Rhom & Haas,

Philadelphia USA) were added into 10 g JME. The mixture was heated at 50-70°C. Then, 5.78 g H_2O_2 35% were added drop by drop into the mixture at temperature that was keeped at 50-70°C (± 5°C). The mixture was stirred and the reaction was held 12-36 hours. The mixture was neutralyzed with saturated $NaHCO_3$ solution. The upper layer (contained JMEE) was washed with aquadest repeatly. JMEE was weighed and analyzed for iodine, oxyrane and peroxide number. Fuctional group analysis was done using FTIR.

The data were analyzed using RSM in Modde 5. The method was used to find out the optimum condition for epoxidation and investigate the influence of reaction time, temperature, and catalyst concentration on oxyrane number. Oxyrane number is shown by color line in three dimensions curve. The optimum condition was validated by holding the reaction on optimum temperature and catalyst concentration with variable time.

RESULT AND DISCUSSION

Table 1 presents the quality properties of JME and JMEE. According to Table 1, iodine number in the epoxide was 5-6 times smaller than the *jatropha curcas* oil methyl ester's, whereas oxyrane number was enhanced 84 times from initial condition. This proved that oxidation was held, one of them was the formation of oxyrane ring, but the oxyrane number that was formed is lower than soybean oil epoxide's, namely ± 6.9% (Petrovic *et al.* 2001).

Table 1. Quality properties of JME and JMEE

Parameter	JME	JMEE (12 hr, 70 °C, 3.0%)
Iodine number (g I_2/100 g)	97.2574	17.46
Oxyrane number (%)	0.04	3.38
Peroxide number (eq/1000g)	14.0825	21.85

Since the low oxyrane number, it is estimated that not all of the double bond in *jatropha curcas* oil methyl ester was converted into its epoxide. This was supported by IR spectrum of *jatropha curcas* oil methyl ester and its epoxide. The other probability, the low oxyrane number was caused by excessive moles of H_2O_2, so the H_2O_2 will cause the opening of oxyrane ring of the epoxide. Beside that, the ring opening was estimated to be caused by the catalyst that is cation exchange acid resin. According Petrovic *et al.* (2001), the opening of oxyrane ring of the epoxide are shown in figure 1.

The influence of temperature and amberlite IR-120 concentration at 12 hours (Figure 2a), 24 hours (Figure 2b), and 36 hours (Figure 2c) reaction time shows that the oxyrane number would enhance when the temperature and the catalyst concentration were enhanced, but the increase of oxyrane number at 12 and 24 hours reaction time were larger than at 36 hours reaction's. The highest increase of oxyrane ring was shown by the reaction at 12 hours, namely 1.38-1.59% to 3.41-3.61%, whereas the reaction at 24 hours showed oxyrane number 1.95-2.07% to 3.16-3.28%. The lowest increase of oxyrane number was shown by reaction at 36 hours, namely 2.42-2.52% to 2.93-3.03%.

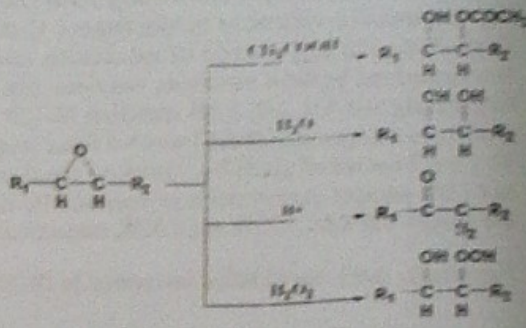

Figure 1. The opening of oxyrane ring of the epoxide (Petrovic *et al.* 2001).

The using of long reaction time (36 hours) caused the enhanced acetic acid concentration as side product of the reaction. The excessive acetic acid can cause the opening of oxyrane ring, so the oxyrane number will be lower (Figure 1.).

The influence of reaction time and catalyst concentration at 50°C (Figure 3a), 60°C (Figure 3b), and 70°C (Figure 3c) shows that the oxyrane number would enhance when the catalyst concentration was enhanced and the raction time was lengthened, but the increase of oxyrane number at 50 and 60°C were higher than at 70°C's. The oxyrane number at 50, 60, and 70°C were 1.38-1.52% to 3.35-3.55%; 2.00-2.11% to 3.14-3.25%, and 2.53-2.64% to 2.85-2.96%, respectively.

The using of high temperature accelarated the oxidation so oxygen binding at double bond was enhanced. According to Paoletti & Kritchevsky (1969), epoxidation mechanism involves the addition of oxygen atom from peroxy acid radical into double bond to form oxyrane compound.

The influence of temperature and time at 1.0%, 2.0%, and 3.0% amberlite IR-120 catalyst concentration is shown by Figure 4. Figure 4a shows that oxyrane number was enhanced from 1.38-1.50% to 2.39-2.50% with higher temperature and longer reaction time, when the catalyst concentration was 1.0%. At 2.0% and 3.0% catalyst concentration (Figure 4b and 4c), the same phenomenons are

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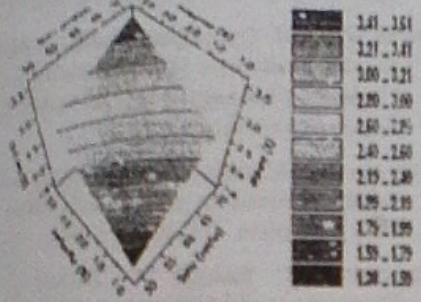
shown. The higher temperature and the longer reaction time caused the increase of oxyrane number to the temperature was ±64°C and the reaction time was ±30 hours with oxyrane number 2.76-2.85% and these values decreased when the temperature was enhanced to 70°C at ±36 hour with oxyrane number 2.67-2.76% (Figure 4b), whereas Figure 4c shows that oxyrane number increased to ±60°C in ±25 hour reaction time, with oxyrane number 3.22-3.28%, and these values decreased at 70°C in 36 hours reaction time, with oxyrane number 2.95-3.02%.



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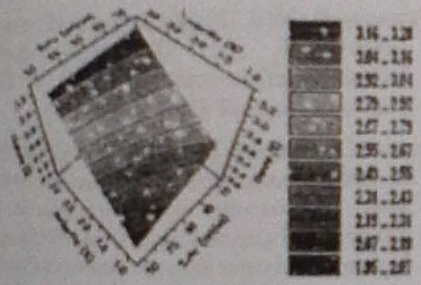

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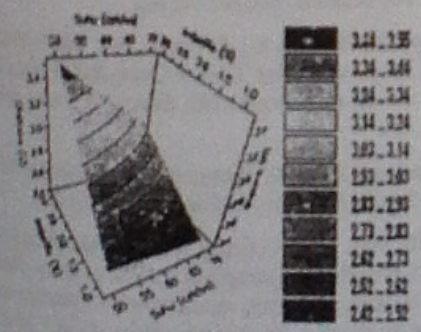
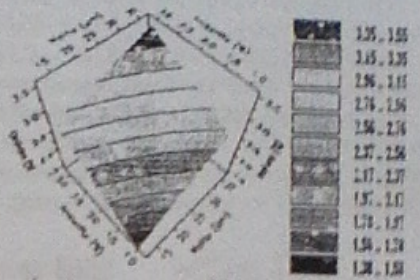


(c)

Figure 2. Curve of influence of temperature and amberlite IR-120 concentration on oxyrane number at reac-tion time (a) 12 hours, (b) 24 hours and (c) 36 hours

At higher catalyst concentration, the increase of oxyrane number was significant in initial condition only, but when the reaction continued, the oxyrane number decreased. It was estimated that H^+ ion that was released by catalyst caused this phenomenon. Catalyst can accelaraie the reaction, but the higher H^+ ion concentration caused the opening of oxyrane ring (Figure 1.).



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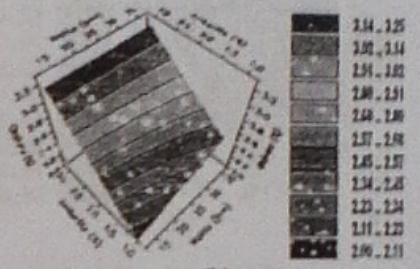

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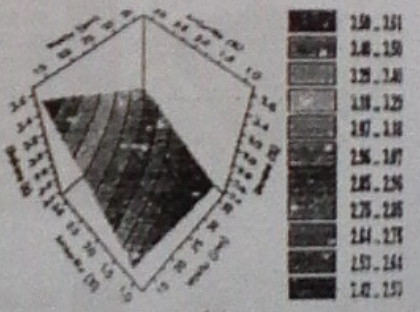

(c)

Figure 3. Curve of influence of reaction time and amberlite IR-120 concentration on oxyrane number at temperature (a) 50°C, (b) 60°C and (c) 70°C

The optimum condition for epoxidation that was obtained using Modde 5. The result were time and temperature reaction and amberlite IR-120 concentration at 12 hours, 70 °C, and 3.0%, respectively, with oxyrane number 3.61%. This point is higher than the oxyrane number that was obtained with catalyst zeolite (1.33%) (Diana 2007) and bentonite (1.36%) (Wahid 2007). The result of validation gave the maximum oxyrane number at 12

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hours reaction time, namely 3.95%. Therefore, the optimum condition that was obtained from Modde 5 is valid.

Analysis of variant and quadratic coefficient assay showed that $P \leq 0.05$, namely 0.001. So, in this research, all of the variables gave the significant influence on oxyrane number. This is supported by three dimensions curve in Figure 2, 3, and 4.

The obtained model for oxyrane number (ON) was $ON = 2.740545 + 0.115490x + 0.157172y + 0.506617z - 0.332287x^2 - 0.118933z^2 - 0.127108yz$, with R^2 and Q^2 0.662 and 0.482 respectively, with x = time, y = temperature, and z = amberlite IR-120 concentration. The regression coefficient of the model is low due to the difficulty in controlling the uniformity of the temperature in all running.

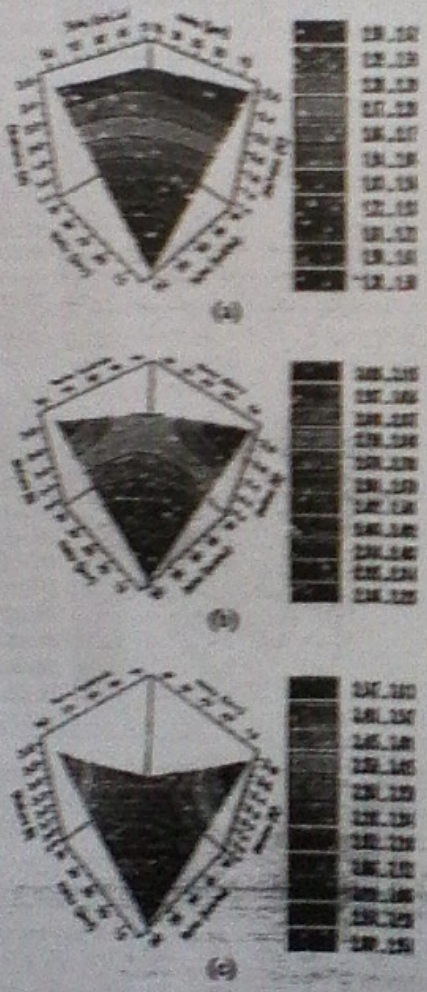

Figure 4. Curve of influence of time and temperature reactions on oxyrane number at amberlite IR-120 concentration (•) 1.0%, (b) 2.0% and (c) 3.0%

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KINETIC STUDY OF *Jatropha curcas* L. METHYL ESTER EPOXIDATION REACTION WITH AMBERLITE IR-120 CATALYST

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ABSTRACT

Phtalate compounds are commonly used as plasticizer in plastic industry. However, these compounds can evaporate from plastic after a period of time so they are harmful for human health and are not environmentally sound. Epoxidized oil such as epoxidized soybean oil have been studied and used as a more safe and green substituent for these compounds.

Jatropha curcas L. contains about 40–60% of oil, which has been utilized as biodiesel. The plasticizing ability of the epoxide derived from this oil, however, has not been studied yet. A preliminary research in order to study the epoxidation kinetics of *Jatropha curcas* L. methyl ester (JME) had been accomplished. We first esterified and transesterified *Jatropha*'s oil into JME, then mixed the product with percarboxylic acid in toluene at 50, 60, and 70 °C for 12 hours, using 3% of ion-exchange acid-resin Amberlite IR-120 as the catalyst. The iodine and oxirane number of the mixture were analyzed every 3 hours to determine the reaction order (n), reaction rate constants (k), and activation energy (E_a). The results showed first order kinetics with k values of 6.24×10^{-2}, 10.17×10^{-2}, and 16.74×10^{-2} hour^{-1} at 50, 60, and 70 °C, respectively, and E_a of 45.43 kJ mol^{-1}.

Keywords: kinetic study, plasticizer, epoxidized oil, *Jatropha curcas* L., epoxidation, methyl ester, Amberlite.

INTRODUCTION

Oil-based plasticizer from phtalic groups is commonly used as plastic additive to give flexibility and endurance. Dibutyl phtalate, dimethoxyethyl phtalate, di-(2-ethylhexyl) phtalate, and dioctyl phtalate are several compounds often utilized. However, according to EPA's Toxic Release Inventory, phatalate compounds are toxic, and since 2006, The European Union has banned their utilization as plasticizer (Tullo 2005), because they are easily migrate to the plastic surface and then released to the environment.

An alternative plasticizer which is biodegradable and renewable is vegetable-oil-based plasticizer, such as from soybean oil (43%), sunflower oil, rice oil, and menhaden oil (Chou & Lee 1997, Supriyadi 1992, and Hazimah *et al.* 2000). Methyl ester from palm oil has also been investigated as plasticizer raw material (Wibowo *et al.* 2003). To be used as plasticizer, vegetable oil or its methyl ester has to be epoxidized. Organic peracid produced *in situ* from carboxylic acid and hydrogen peroxide is usually used as the oxygen donor to the oil's or its methyl ester's double bonds (Campanella & Baltanas 2005).

Today in Indonesia, vegetable oil has been utilized in cosmetic and food industry and as an alternative fuel (biodiesel), but it has not yet been used as plasticizer. Meanwhile in Europe and Japan, oil-based plasticizer has substituted phtalate for medical utilities. In accordance with Indonesian government's policy to promote mass production of biodiesel until 2010, the vegetable oil from *Jatropha curcas* L. seed is being increasingly studied now.

Oil compose 40–60% of *Jatropha*'s seed (Shah 2003). The physical and chemical properties of this oil are similar to soybean oil. Gubitz *et al.* (1998) in Marsurung (2005) reported that unsaturated fatty acids dominate this oil, especially oleic acid (34.3–45.8%) and linoleic acid (29.0–44.2%). This fact suggested that *Jatropha*'s oil has a potency to be epoxidized as plasticizer, besides being utilized as biodiesel.

Sugita *et al.* (2007) had reported optimization of *Jatropha*'s methyl ester (JME) epoxidation reaction using *Modde 5.0* software. The reaction time and temperature were varied to 12, 24, and 36 hours and 50, 60, and 70 °C, respectively, by using 1, 2, and 3% of Amberlite IR-120 as the catalyst. The optimum oxirane number (3.61%) was obtained when the reaction was carried out for 12 hours, at 70 °C, and with 3% of catalyst. The optimum oxirane number obtained from the research was quite similar to this result, that was 3.95%.

In this research, the kinetics of the JME's epoxidation reaction at that optimum condition was studied by measuring the decrease of iodine number and the increase of oxirane number at every 3 hours. The kinetic parameter obtained included reaction order (n), reaction rate constant (k), and activation energy (E_a).

METHODS

JME Epoxidation and Kinetics Study

JME epoxide (JMEE) was synthesized by modifying Rios (2003) procedure, using the apparatus shown at Figure 1. Solution I, 100 g of JME (esterified and transesterified at Puspiptek Serpong from *Jatropha*'s seed obtained from Jember, East Java) mixed with 8 mL of glacial acetic acid, 29 mL of toluene, and 3 g of Amberlite IR-120 catalyst

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[3%(w/w), *Rhom & Haas* (Philadelphia, USA)], was poured into a three-neck flask. Then solution II, 57.8 g of 35% H_2O_2, was added slowly into the flask from a separatory funnel, and the mixture was stirred well for 12 hours at a relatively constant temperature (50, 60, and 70 °C).

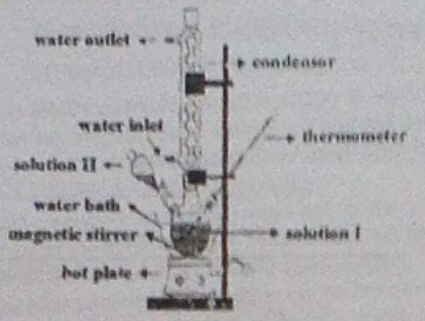

Figure 1 JME epoxidation apparatus

Every 3 hours, 7 mL of aliquot was taken from the reaction mixture, neutralized with sodium bicarbonate solution, washed several times with distilled water, and analyzed for its iodine and oxirane number. The reaction order (n) was determined from the highest determination coefficient (r^2) amoag 3 type of regression curves: iodine number (I), ln I, and $1/I$ versus reaction time (t: 0, 3, 6, 9, and 12 hours), showing zero, first, and second order kinetics, respectively. The reaction rate constans, k, were determined from gradients of the selected curve type. Finally, by plotting ln k against $1/T$ at 3 different temperatures (50, 60, and 70 °C), the activation energy (E_a) was calculated.

Analysis of JME and JMEE

Analysis of Functional Groups and Methyl Ester Composition. Functional groups in JME and JMEE were analyzed with Fourier transform infrared (FTIR) spectrophotometer Shimadzu 8400 in Chemistry Department, UPI (Chou & Lee 1997; Gan *et al.* 1992, Rangrajan & Harvey 1995, Rios 2003). The methyl ester composition in JME was also examined by using gas chromatography-mass spectrometry (GC-MS) Shimadzu AOC-20i *autoinjector* in Chemistry Department, UIN Syarif Hidayatullah with

Column type	: MPS
Column length	: 30 m
Detector	: Electron Impact (EI)
Carrier gas	: He
Flow rate	: 1.5 mL/minute

Iodine Number (AOAC 1984). Into *ca.* 0.5 g of JME in a 250 mL Erlenmeyer flask, 10 mL of chloroform and 25 mL of Hanus solution (IBr) were added. The mixture was allowed to stand in the dark for about 60 minutes. Afterwards, 20 mL of 10% KI solution was added and the flask was stoppered immediately. After being shaked for a moment, the mixture was titrated with 0.1 N $Na_2S_2O_3$ solution until it became faint yellow. Then, 1% of amylum indicator was added and the titration was continued until the dark blue colour disappear. The determination of blank was also carried out. The iodine number was calculated as the average of 3 replicates, using the following equation:

$$\text{Iodine value} = \frac{(\text{mL blank} - \text{mL sample}) \times N_{\text{tio}} \times 12.69}{\text{sample weight (g)}}$$

Oxirane Number (AOCS 1993). Into a 50 mL Erlenmeyer flask, (0.3–0.5) g (± 0.0001 g) of JME was dissolved in 10 mL of glacial acetic acid. Five drops (maximum 0.1 mL) of violet crystal indicator was then added and the mixture was titrated by 0.1 N HBr solution until the colour turned into bluish green for about 30 seconds. The oxirane number was calculated as an average of 3 replicates, as follows:

$$\text{Oxirane number} = \frac{(V \times N)_{HBr} \times 1.60}{\text{sample weight (g)}}$$

RESULTS AND DISCUSSIONS

Analysis of JME and JMEE

The GC-MS chromatogram showed that JME contained several methyl ester compounds (Table 1). Methyl palmitoleate, a monounsaturated methyl ester, was the predominant one (54.24%). The presence of unsaturated methyl ester compounds in JME was proved by its iodine number, 97.26 g I/100 g (Table 2). After epoxidation, this value was significantly decreased to 17.46 g I/100 g. In the other hand, the oxirane number of JMEE became 84 times higher than JME (0.04 to 3.38%). These results showed that JME had been epoxidized, but the oxirane number was relatively low compared with epoxidized soybean oil (ESO), i. e. 6.9% (Petrović *et al.* 2001).

Table 1 Methyl ester compounds in JME

Compound	Composition (%)
Methyl octanoate	2.24
Methyl caprate	0.28
Methyl laurate	2.61
Methyl tetradecadienoate*	0.09
Methyl 5-octadeceuoate*	0.29
Methyl myristate	4.77
Methyl pentadecanoate	0.80
Methyl 7,10-hexadecadienoate*	0.52
Methyl palmitoleate*	54.24

(*): unsaturated methyl esters.

Table 2 Characteristics of JME and JMEE

Parameter	JME	JMEE (12 h, 70 °C, 3%)
Iodine number (g I/100 g)	97.26	17.46
Oxirane number (%)	0.04	3.38

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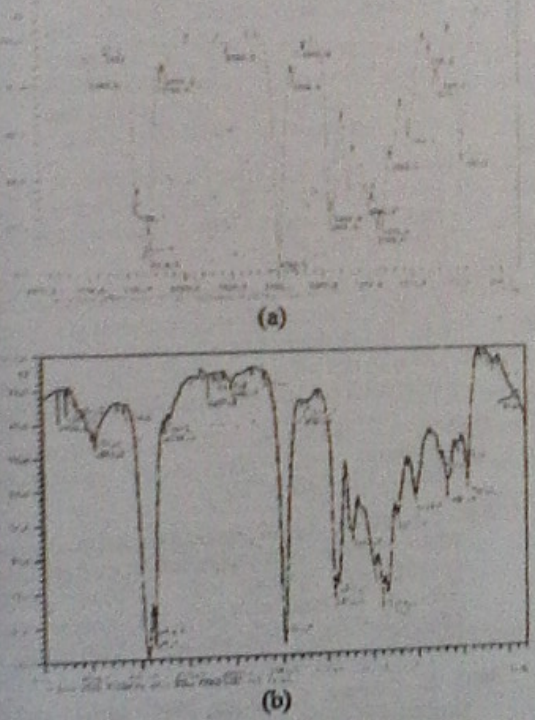
Peroxide number (meq/g) = 21.85

The relatively low oxirane number is supported by the FTIR spectrum (Figure 2). Both spectra showed C–O, C=C, and C=O groups. The C–O peaks of JME were obtained at 879.5, 914.2, and 1245.9 cm^{-1}, the C=C peaks at 1600.8 and 1654.8 cm^{-1}, and the C=O peak at 1743.5 cm^{-1}. For JMEE, the peaks were obtained at 879.5 and 1245.9 cm^{-1}; 1604.7 and 1627.8 cm^{-1}; and 1739.7 cm^{-1}, respectively. It was predicted that the double bonds of JME were not only epoxidized, but also oxidized into peroxides, as shown by the JMEE's peroxide number, 21.85 meq/g (Table 2). Epoxide ring opening in the acid condition during the epoxidation reaction was also possible. Petrović *et al.* (2001) suggested several ring-opening mechanisms (Figure 3), which could be caused by an excess of H_2O_2 or by the cation-exchange acid-resin type of Amberlite IR-120.



(a)

(b)

Figure 2 FTIR spectrum of JME (a) and JMEE (b).

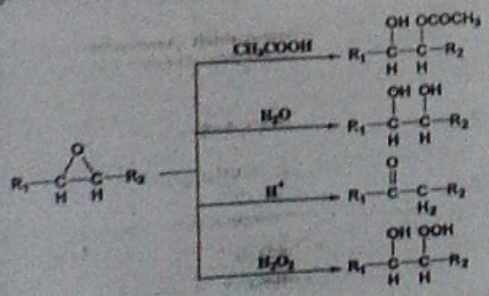


Figure 3 Epoxide ring-opening mechanisms.

Epoxidation Kinetics of JME

Epoxidation reaction rate can be expressed as the decreasing rate of iodine number or the increasing rate of oxirane number. In our study, we used the first expression. It was assumed that all double bonds of JME was converted into epoxides.

The iodine number (I), ln I, dan $1/I$ from all aliquots for the 3 temperature studied were given in Table 3. By plotting these values as y-axis to time t as x-axis, the regression curves for zero, first, and second order for each temperature were obtained respectively. The equations were summarized in Table 4. The highest determination coefficient (r^2) was obtained for the first order equation: 0.9975, 0.9931, and 0.9959 at 50, 60, dan 70 °C, respectively. This result showed that the *in situ* JME's epoxidation in this research followed first order reaction kinetics.

Table 3 Iodine number (I), ln I, dan $1/I$ at various time

Temp. (°C)	t (hours)	I (g I/100 g)	ln I	$1/I$
70	0	75.18	4.32	0.01
	3	42.79	3.76	0.02
	6	27.43	3.31	0.04
	9	17.70	2.87	0.06
	12	9.48	2.25	0.11
60	0	72.15	4.28	0.01
	3	53.53	3.98	0.02
	6	37.58	3.63	0.03
	9	27.05	3.30	0.04
	12	22.06	3.09	0.05
50	0	77.33	4.35	0.01
	3	61.59	4.12	0.02
	6	52.33	3.96	0.02
	9	43.67	3.78	0.02
	12	36.00	3.58	0.03

Table 4 Determination coefficient of zero, first, and second order equation at 50, 60, and 70 °C

Temp. (°C)	Order	Regression curve equation	r^2
50	0	$y = -3.3527x + 74.301$	0.9772
	1	$y = -0.0624x + 4.3319$	0.9975
	2	$y = 0.0012x + 0.0125$	0.9893
60	0	$y = -4.2219x + 67.806$	0.9541
	1	$y = -0.0917x + 4.2657$	0.9931
	2	$y = 0.0027x + 0.012$	0.9862
70	0	$y = -5.216x + 65.812$	0.9131
	1	$y = -0.1674x + 4.3068$	0.9959
	2	$y = 0.0072x + 0.0035$	0.8900

The reaction rate equation for the first order is expressed as

$$-\frac{dI}{dt} = kI \quad \ldots (3)$$

which can be integrated into

$$\ln\left(\frac{I_o}{I}\right) = kt \quad \ldots (4)$$

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Equation (4) showed that reaction rate constant k can be obtained as the gradient of regression curve between $\ln(I_0/I)$ and t. Figure 5 showed these curves at 50, 60, and 70 °C. The k values were summarized in Table 5, and used to determine the activation energy, E_a, by using logaritmic form of the Arhennius equation:

$$\ln k = \ln A - \frac{E_a}{R}\left(\frac{1}{T}\right) \quad \ldots (5)$$

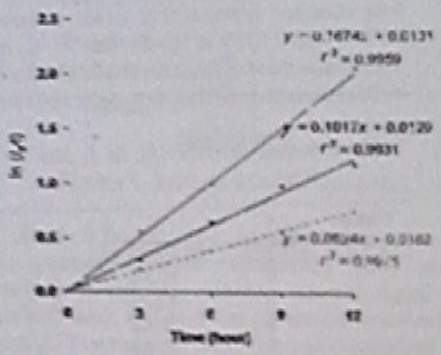

Figure 5 Regression curves between ln (I_0/I) and time t at 50 (▲), 60 (■), and 70 °C (♦).

Table 5 JME's epoxidation reaction rate constants at different temperatures

Temp (°C)	k (hour^{-1})	r^2
50	6.24×10^{-2}	0.9975
60	10.17×10^{-2}	0.9931
70	16.74×10^{-2}	0.9959

Plot of ln k to $1/T$ gave $y = -5464.3x + 14.137$ as the Arrhenius equation with r^2 of 0.9995. From this equation, it was found that the activation energy for JME's epoxidation in this research was 45.43 kJmol^{-1}. This value is lower than the activation energy for *in situ* epoxidation of soybean oil by using peroxoacetic acid in toluene with Amberlite IR-120 as the catalyst, that is 54.7 kJ mol^{-1} (Petrović *et al.* 2001). However, it is still higher then the activation energy of palm oil methyl ester epoxidation, i. e. 37.47 kJ mol^{-1} (Wibowo *et al.* 2003). These results suggested that epoxidation of methyl ester is kinetically better than that of its vegetable oil form. However, palm oil methyl ester is still better than JME to be epoxidized.

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