

## Spectroscopic Analysis of Epoxidised Jatropha Oil (EJO) and Acrylated Epoxidised Jatropha Oil (AEJO)

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### ABSTRACT

The non-edible seed oil of the Jatropha plant is a renewable and sustainable material to produce vegetable oil-based epoxy and epoxy acrylate as raw polymeric material. The objective of this study is to synthesis Jatropha seed oil-based epoxy and acrylate epoxy resins through conventional methods. An epoxy ring of Epoxidised Jatropha Oil (EJO) was formed through an *in-situ* epoxidation process using hydrogen peroxide and formic acid as an oxygen donor and oxygen carrier respectively. Acrylated Epoxidised Jatropha Oil (AEJO) was produced by reacting EJO with acrylic acid with the existence of triethylamine and 4-methoxyphenol as a catalyst and inhibitor respectively. The measured oxirane-oxygen content (OOC) of EJO was 4.99%. The acid value of the AEJO was determined at 4.42 mg KOH/g. Both the EJO and AEJO were characterised by FTIR and <sup>1</sup>H NMR spectroscopies.

*Keywords:* Acid value, acrylated epoxidised jatropha oil, epoxidised jatropha oil, FTIR, NMR, oxirane-oxygen content

### INTRODUCTION

Natural vegetable oil consists of 93-98 wt.% triglyceride and minor components of diglycerides, monoglycerides and phosphoglycerides (Sharmin, et al., 2015). Triglycerides can be defined as ester where three molecules of one or more different types of fatty acids are linked to the alcohol glycerol. Fatty acids are normally made up of 14 to 18 carbon atoms linearly. It

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consists of saturated, monounsaturated and polyunsaturated fatty acids. Belgacem and Gandini (2011, p. 52) found that some of the fatty acids consist of hydroxyl and epoxy groups such as ricinoleic and vernolic acids respectively, and the presence of these functional groups is more reactive but it is rare in naturally produced oil. Therefore, active sites of vegetable oil such as double bonds, allylic carbons, oxiranes and ester groups can be chemically and enzymatically transformed into functionalised polymeric material to increase the reactivity of vegetable oil (Xia & Larock, 2010).

Jatropha Oil (JO) can be used in polymer material synthesis compared with edible oils such as palm oils, soybean oils and sunflower oils because it is abundant and classified as non-edible oil due to the toxic phorbol ester in the seed (Hazmi, et al., 2013; Islam, et al., 2014). *Jatropha curcas* is a flowering plant of family Euphorbiaceae (Martinez-Herrera, et al., 2006; Kumar & Sharma, 2008), genus *Jatropha* and this family contains 175 of the known species (Singh et al., 2008; Karaj & Müller, 2010). It originates from Central America, Africa, South and South East Asia (Martinez-Herrera et al., 2006; Vyas & Singh, 2007; Singh et al., 2008; Zippel, et al., 2010) and can be grown in tropical and sub-tropical zones. Its seed is blackish, thin-shelled and oblong in shape (Kabir, et al., 2009). *Jatropha* seed is a good source of oil where the decorticated *Jatropha* seed consists of 40-60% oil (Makkar, et al., 1998; Openshaw, 2000; Sharma, et al., 1994), 75% unsaturated fatty acid, mainly oleic acid (18:1) and linoleic acid (18:2) at percentages

of 42.8% and 32.8% respectively (Akbar, et al., 2009) and thus it is known as an oleic-linoleic oil.

Highly unsaturated non-edible JO is suitable to be transformed from its less reactive double bond into various *Jatropha*-based derivatives which are more reactive than virgin oil. Due to the low iodine number and high cetane concentration of mature *Jatropha* seed oil, much research had been focused on the potential of *Jatropha* seed oil as a renewable energy resource for biodiesel (Openshaw, 2000; Contran et al., 2013; Sinha, et al., 2015). The performance of trans-esterified *Jatropha* seed oil by using methanol in the presence of an alkali catalyst was achieved and which meets the standard requirements of American and European countries (Gübitz, et al., 1999) and is within the limits of the American Society for Testing and Materials (ASTM) specification for diesel, D 6751 (Kywe & Oo, 2009). In addition, the high content of unsaturated fatty acids makes *Jatropha*-based polyurethane adhesives exhibit better shear strength and good resistance towards water, acid and alkali solvents compared with palm oil based adhesives (Aung, et al., 2014). The transformed JO has been projected as a potent base stock for bio-lubricants at high temperature due to its high flash point property (Sammaiah, et al., 2014).

Epoxidised vegetable oil acts as a vital raw material in the synthesis of polymer derivatives such as acrylates, polyols, polyesters, polyamides and other derivatives via the ring opening process.

Acrylate epoxy resin is usually applied in UV-cured coatings due to its high reactivity of acrylate unsaturated functional groups. Acrylate epoxy is known to be applied in graphic arts, automotive coatings, and floor coatings (Ronda, et al., 2013). In the past, acrylate epoxy resin was produced commercially with petroleum as the raw materials. Utilisation of petroleum-based products over the long term has raised environmental and health hazard concerns, not to mention the depletion of petroleum resources which has attracted the attention of many researchers in the utilisation of plant oil derived polymeric materials (Alam, et al., 2014). For instance, the use of palm oil (Cheong, et al, 2009), soybean oil (Habib & Bajpai, 2011) and tung oil (Thanamongkollit, et al., 2012) in several applications such as adhesives, lubricants, biodiesels, coatings etc. has been studied in recent years. The objective of this study is to synthesis Jatropha-based acrylate epoxy resin by reacting Epoxidised Jatropha Oil (EJO) with acrylic acid in the presence of 4-methoxyphenol and triethylamine.

## MATERIALS AND METHOD

### Materials

Jatropha oil with a composition of oleic acid (41.3%), linoleic acid (34.4%), palmitic acid (12.8%), stearic acid (7.3%) and unsaturated fatty acid (2.7%) was obtained from Bionas Malaysia Sdn. Bhd. Hydrogen peroxide (30%), sulphuric acid (95%) and hydrated magnesium sulphate (99.5%) were purchased from Merck, Germany. Triethylamine was supplied by Fluka, Spain.

Formic acid (98%), acrylic acid (99.5%) and 4-methoxyphenol (99 %) were obtained from Across Organic, New York.

### Epoxidation of Jatropha Oil

The *in-situ* epoxidation of JO was carried out in a three-necked round bottom flask fitted with a mechanical stirrer, a thermometer and a dropping funnel. The JO (100 g) was charged into the flask followed by formic acid (11.66 g) and the flask was continuously stirred in a water bath at 45-55°C. The molar ratio of the double bond of oil to formic acid and hydrogen peroxide was 1: 0.6: 1.7. A corresponding quantity of hydrogen peroxide (73.25 g) was added into the reaction mixture at such a rate as to reach a dropping time of 30 minutes. The dropping rate of hydrogen peroxide should be slow enough to prevent a sudden increase in reaction temperature. Precaution was taken to avoid overheating due to exothermic reaction. After that, the mixture was heated to 60°C and further stirred for 4 hours. The cooled EJO was washed three times with distilled water in order to remove the remaining acid. The aqueous layer was removed and a little bit of anhydrous MgSO<sub>4</sub> was added and then left overnight.

### Acrylation of Epoxidised Jatropha Oil

Previously prepared EJO (100 g) was charged in a three-necked flask equipped with a mechanical stirrer, a thermometer and a dropping funnel. The flask was placed in a silicone oil-bath at room temperature and stirring at 500 rpm. After that, triethylamine

(2 wt.% of EJO and acrylic acid) and 4-methoxyphenol (1wt.% of EJO and acrylic acid) were added to the mixture at 110°C and stirred for 15 minutes. Under continuous stirring, a calculated amount of acrylic acid (3.18 g) was added drop by drop to the mixture for about 30 minutes. The molar ratio of EJO to acrylic acid (EJO: CH<sub>2</sub>=CHCOOH) was 1:1. After the addition of acrylic acid, the reaction was stopped until a constant acid value (less

than 5 mg KOH/g) was achieved. Cooled Acrylated Epoxidised Jatropha Oil (AEJO) was washed three times with distilled water in order to remove the remaining acid. The aqueous layer was removed and the AEJO resin underwent rotary evaporation to remove excessive acrylic acid until a clear and viscous product was obtained. A little bit of anhydrous MgSO<sub>4</sub> was added and then left overnight. The reactions of epoxidation and acrylation are shown in Figure 1.

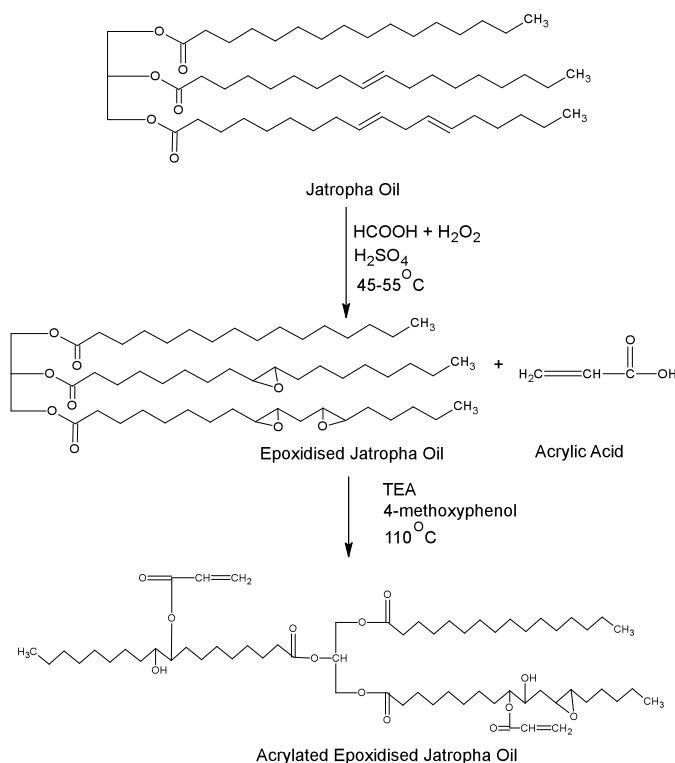


Figure 1. Epoxidation of JO and acrylation of EJO reactions

### Fourier Transform-Infrared Spectroscopy (FT-IR) Analysis

The Fourier Transform-Infrared Spectroscopic (FT-IR) spectra of the received

JO, EJO and AEJO were determined by means of a Perkin Elmer Model 1650 FTIR spectrometer using KBr pellets.

### <sup>1</sup>H Nuclear Magnetic Resonance (<sup>1</sup>H NMR) Analysis

The received JO, EJO and AEJO were sent for <sup>1</sup>H NMR analysis by utilising the model Varian NMR System. <sup>1</sup>H Nuclear Magnetic Resonance (<sup>1</sup>H NMR) spectra were obtained with a Varian Unity INOVA Spectrometer. Chemical shifts ( $\delta$ ) were recorded in ppm relative to tetramethylsilane (TMS). Deuterated chloroform (CDCl<sub>3</sub>) was used as a solvent.

### RESULTS AND DISCUSSION

The composition of unsaturated fatty acid in JO and other plant oils is shown in Table 1. The total unsaturated fatty acid of JO is higher than the commercial palm oil at 78.9%. Its high composition of oleic acid (43.4%) and linoleic acid (34.4%) are comparable to sunflower oil. JO is known as an oleic-linoleic oil.

Table 1  
*Unsaturated fatty acids of plant oils*

Fatty Acid	Percentage (%)			
	Jatropha Oil	Palm Oil	Soybean Oil	Sunflower Oil
Palmitoleic (C16/1)	1.4	-	0.1	0.8
Oleic (C18/1)	43.1	43.4	23.4	20.6
Linoleic (C18/2)	34.4	13.2	53.2	66.2
Linolenic (C18/3)	-	-	7.8	0.8
Total Unsaturated	78.9	56.6	84.5	88.4

Adapted from (Meyer et al., 2008)

One of the important properties in the characterisation of epoxy vegetable oil is the determination of the oxirane-oxygen content (OOC), in order to ensure the epoxy groups are present in the products. Epoxy resins with a high OOC are desired in the production of polymer. EJO was synthesised through *in-situ* reaction. Peroxyformic acid was formed by formic acid accepting oxygen atoms from an oxygen donor, hydrogen peroxide as shown in Figure 2. Unstable peroxyformic acid then attacked the double bonds of the JO, causing the ring formation, and resulting in EJO as shown in

Figure 3. An OOC test was conducted by the direct method with hydrobromic acid (HBr) solution in acetic acid according to the ASTM D1652-97 Test Method A Standard to form the following expression:

$$\text{OOC Percentage} = (V-B) \times N \times 1.60 / W \quad [1]$$

where V is the volume of HBr solution used for titration of the sample in ml, B is the volume of HBr solution used for titration of the blank in ml, N is the normality of HBr solution in acetic acid, and W is the weight of sample (in gram).

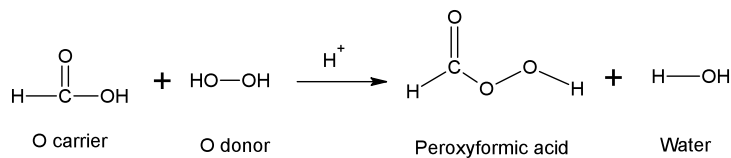


Figure 2. Formation of peroxyformic acid

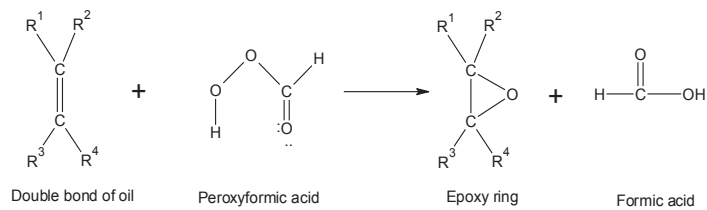


Figure 3. Formation of epoxy ring

The OOC was determined every half an hour and the reaction was stopped when the OOC dropped after achieving a maximum value. The epoxy content of the synthesised EJO was 4.99% which is higher than the EJO that was synthesised from chemo-enzymatic treatment with an

OOC percentage of 4.60% (Togi, 2010). According to the recorded OOC values, the reaction achieved the highest OOC value within 4h as shown in Figure 4. Along with the reaction, the oxirane content was increased due to the formation of epoxy rings (Hazmi et al., 2013).

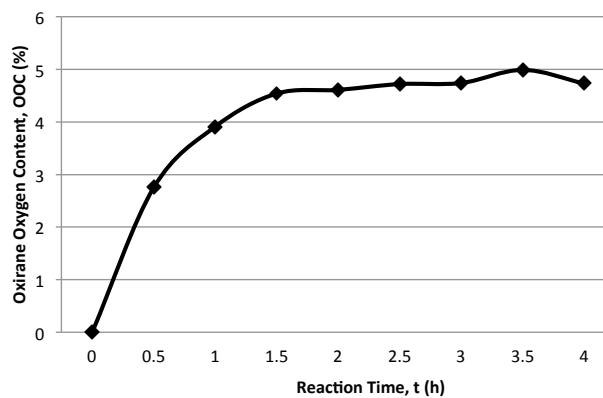


Figure 4. A graph of OOC percentage versus reaction time

In the acrylation process, triethylamine and 4-methoxyphenol acted as catalyst and gel inhibitor respectively. The lone pair electrons on the nitrogen atom of triethylamine will attack the unstable epoxy group and thus a complex intermediate will be formed. The nucleophile of acrylic acid substituted triethylamine and formed a stable ester bond through a nucleophilic substitution reaction. As a result, AEJO was

produced. Figure 5 shows the mechanism of the acrylation reaction. An acid value (AV) was measured along with the reaction to indicate the mass of potassium hydroxide (KOH) in milligrams that was required to neutralise one gram of product. An AV test was carried out according to the ASTM D4662-98 Test Method A Standard by measuring the acidic constituents in polyols and other materials of high acidity

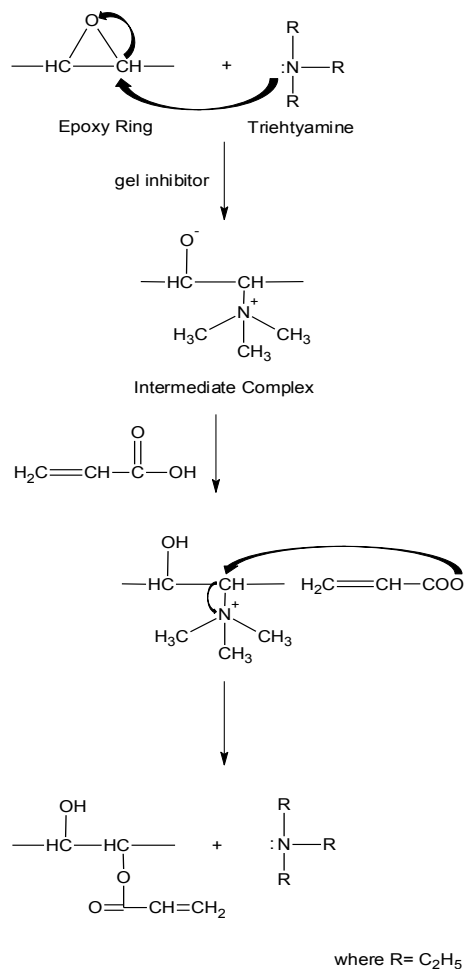


Figure 5. Mechanism of acrylation reaction

that are soluble in mixtures of toluene and ethyl alcohol. The AV was calculated by the following expression:

$$AV = (V-B) \times N \times 56.1 / W \quad [2]$$

where V is the volume of KOH solution used for titration of the sample in ml, B is the volume of KOH used for titration of the blank in ml, N is the normality of KOH solution in ethanol, and W is the weight of sample used in gram.

The rate of addition of acrylic acid and the temperature of the reaction must not exceed 110°C in order to prevent the formation of gel during the reaction. The rate of stirring should be increased when the viscosity of the mixture increases. The acid value of the synthesised AEJO was 4.42 mg KOH/g. Table 2 shows the OOC percentage and AV of the samples. The AV decreased over time for each sample due to the process of acrylation.

Table 2  
OOC percentage and acid value of samples

Sample	Oxirane-Oxygen Content Percentage (%)	Acid Value (mg KOH/g)
EJO	4.99	23.86
AEJO	1.56	4.42

### Fourier Transform Infrared (FTIR) Spectroscopy Analysis

Fourier Transform Infrared (FTIR) spectroscopy is a fast, non-destructive technique that is used widely in the characterisation of lipids because of the absorption bands of the functional groups within the lipids at the infrared region in the electromagnetic spectrum. The FTIR spectra of JO, EJO and AEJO are shown in Figure 6. The spectrum of JO showed a CH stretching of saturated fatty acid backbone at 2929 cm<sup>-1</sup> and 2858 cm<sup>-1</sup> which represent a stretching band of terminal methyl group (CH<sub>3</sub>) and methylene proton

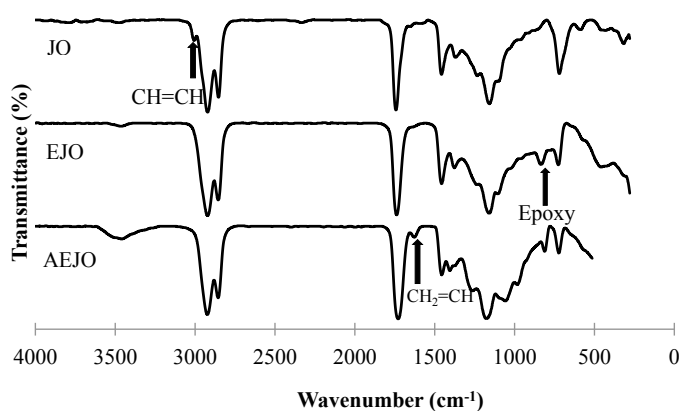


Figure 6. FTIR spectra of received JO, EJO and AEJO



(CH<sub>2</sub>) of triglyceride molecule respectively. The peak around 1749 cm<sup>-1</sup> is attributed to the carbonyl group (C=O) of triglyceride backbone. A significant functional group of unsaturated fatty acid was displayed at 3006 cm<sup>-1</sup> which indicated the vinyl proton of unsaturation double bond (CH=CH). For EJO FTIR analysis, the alkene double bond of JO at 3006 cm<sup>-1</sup> disappeared due to the formation of epoxy rings. This is supported by many researches done on epoxidation of vegetable oil (Adhvaryu & Erhan, 2002; Dinda, et al., 2008; Hazmi et al., 2013). A C-O-C oxirane group was observed at 830-850 cm<sup>-1</sup> and the small intensity at 830

cm<sup>-1</sup> represented the most significant peak indicating the presence of the oxirane group. These findings are consistent with the study by (Habib & Bajpai, 2011). In the FTIR spectrum of the AEJO, the diminishing small peak at 830 cm<sup>-1</sup> represented the open ring reaction and indicated that not all epoxy groups have been consumed during the acrylation reaction. The OH bond showed a broad peak at 3476 cm<sup>-1</sup> and a CH<sub>2</sub>=CH stretch at an intensity of 1637 cm<sup>-1</sup> indicated the formation of AEJO. The results are similar with those of (Rengasamy & Mannari, 2013). Table 3 represents the FTIR absorption peaks of JO, EJO and AEJO.

Table 3  
FTIR absorption peaks of JO, EJO and AEJO

Functional Group	Absorption Peak (cm <sup>-1</sup> )		
	JO	EJO	AEJO
CH=CH	3006	-	-
CH stretch	2929, 2858	2923, 2857	2925, 2858
C=O	1749	1738	1737
CH scissoring band	1465	1453	1453
CH symmetric band	1381	1373	1371
C-C-O	1174	1163	1160
C-O-C oxirane group	-	830-850	830
OH (strong)	-	3473	3473
CH <sub>2</sub> =CH	-	-	1636

### **<sup>1</sup>H Nuclear Magnetic Resonance (NMR) Spectroscopy Analysis**

<sup>1</sup>H NMR spectroscopy was utilised to verify the structures of the received JO, EJO and AEJO. The spectra of the samples are shown in Figure 7. In <sup>1</sup>H NMR spectrum of JO, a triplet peak at chemical shift, δ of 0.80-0.88

ppm is represented by the terminal methyl protons (-CH<sub>3</sub>) of fatty acid. Chemical shift at 1.19- 1.25 ppm, 1.23-1.26 ppm, 2.21-2.28 ppm and 1.53-1.69 ppm are revealed as the methylene protons adjacent to terminal proton (-CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), methylene protons in saturated fatty acid chain (-CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>-), methylene protons adjacent to carbonyl

group ( $\alpha,\beta$ -proton) ( $-\text{OC}=\text{OCH}_2\text{CH}_2-$ ) respectively. Methylene protons of unsaturated double bonds ( $-\text{CH}=\text{CH}-$ ) were assigned to the  $\delta$  at 5.24-5.32 ppm and the  $\delta$  at 5.19-5.24 ppm and 4.06-4.29 ppm represented the protons in the glyceride backbone of JO ( $\text{CH}(\text{CH}_2\sim)_2\text{OCO}\sim$  and  $\text{CH}_2(\text{CHOCO}\sim)\text{OCO}\sim$ ). For the  $^1\text{H}$  NMR spectrum of EJO, the disappearance or diminishing of unsaturation at 5.24-5.32

ppm and the presence of epoxide group with a  $\delta$  at 3.06-3.07 ppm is determined. This confirmed the formation of epoxy ring in EJO. This finding is similar with those of study worked on the epoxidation of soybean oil where a new peak was found at 3.0 ppm and diminishing of peaks at 5.2 ppm (Habib & Bajpai, 2011). A small signal at chemical shift of 1.71-1.99 ppm is revealed as the methylene proton adjacent

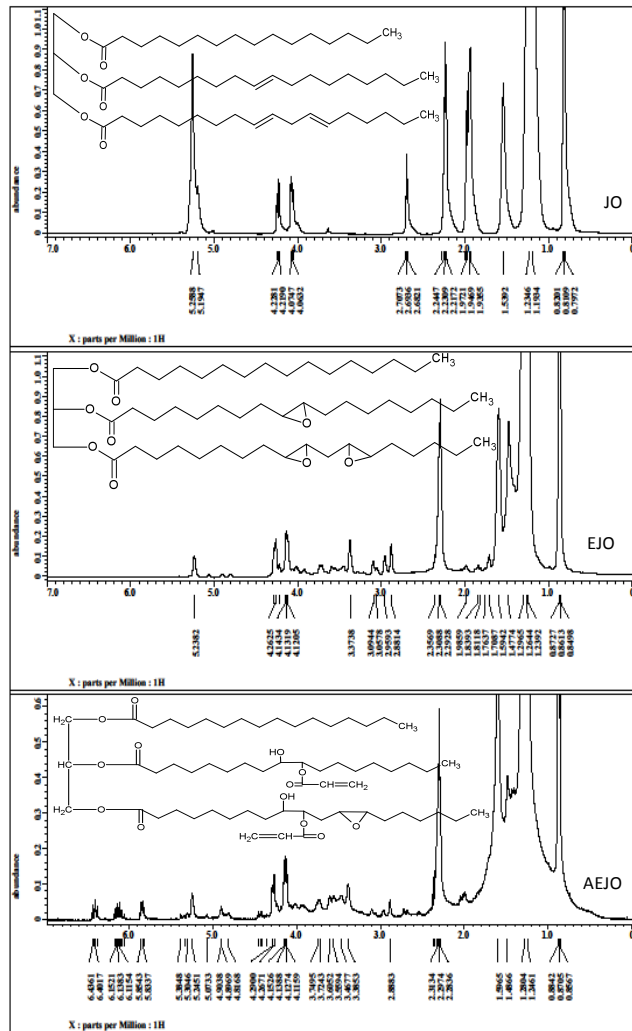


Figure 7.  $^1\text{H}$  NMR spectra of received JO, EJO and AEJO

to epoxy group (Epoxy-CH<sub>2</sub>-) whereas 2.88-2.96 ppm is represented as the methylene proton adjacent to two epoxy groups (Epoxy-CH<sub>2</sub>-Epoxy). On the other hand, the formation of an acrylate group in the AEJO was proved by the new peaks at 5.83-6.43 ppm assigned to protons of acrylate ester (-OCO-CH=CH<sub>2</sub>). The formation of hydroxyl proton (-OH) due to the opening of epoxy ring is attributed to chemical shift of 2.88 ppm, similar to findings of earlier studies (Téllez, et al., 2009). Methylene proton adjacent to hydroxyl proton (-CH-OH) appears at 3.37-3.61 ppm which is a more de-shielded region compared with methylene proton which is adjacent to the epoxy group. This is because these methylene protons are adjacent to oxygen atom of hydroxyl group which experiences stronger anisotropy effect compared with methylene proton adjacent to the epoxy group (Salih et al., 2015).

## CONCLUSION

The AEJO with an acid value of 4.42 mg KOH/g was successfully synthesised from JO through two significant reactions, epoxidation and acrylation. From <sup>1</sup>H NMR spectra, the disappearance of methylene proton in unsaturated double bonds of JO at chemical shift of 5.24-5.32 ppm and the presence of epoxide group at 2.7-2.9 ppm confirmed the conversion of EJO from JO. On the other hand, the formation of an acrylate group and hydroxyl group at 5.83-6.43 ppm and 2.88 ppm respectively

represent epoxy ring opening and grafting of acrylate groups.

## RECOMMENDATION

The green acrylate epoxy resin can be used in the coating and adhesive industry which is able to reduce environmental impacts. The application of AEJO as anticorrosion coating could form the basis of further study in order to increase the corrosion performance of the coating on a mild steel substrate.

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