

## SYNTHESIS AND CHARACTERIZATION OF BIOBASED EPOXIDIZED EDIBLE OILS

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**Abstract:** Nowadays, major pollutions present in the environment are produced by non-degradable substances. Eventually, the use of non-degradable products will increase carbon footprint in the atmosphere. In this study, modifications of five vegetable oils into biobased epoxides are reported. The oils used in this study were commercial palm oil, rice bran oil, canola oil, soybean oil and sunflower oil. The vegetable oils were refluxed using formic acid and hydrogen peroxide at temperature ranges of 45 to 50°C and 60 to 65°C for seven hours in order to change the chemical structure of carbon-carbon double bonds into oxirane rings. The mixture was then processed by liquid-liquid extraction to separate epoxide oil from liquid that was present during the reflux process. The obtained oils were then analyzed using ATR-FTIR and the presence of oxirane rings were observed at the wavelength  $\nu=1080.79\text{ cm}^{-1}$  and  $836.10\text{ cm}^{-1}$  for epoxidized palm oil,  $\nu=1107.14\text{ cm}^{-1}$  and  $841.11\text{ cm}^{-1}$  for epoxidized rice bran oil,  $\nu=1050.13\text{ cm}^{-1}$  and  $850.35\text{ cm}^{-1}$  for epoxidized canola oil,  $\nu=1083.99\text{ cm}^{-1}$  and  $825.03\text{ cm}^{-1}$  for epoxidized soybean oil and  $\nu=1095.57\text{ cm}^{-1}$  and  $820.16\text{ cm}^{-1}$  for epoxidized sunflower oil, respectively. Additionally, an absorption band at  $\nu=1462\text{ cm}^{-1}$  was observed in all oil samples, indicating the presence of C-C oxirane. In future, these epoxidized oils can serve as a great potential as new starting materials for the synthesis of lipid biopolymers.

Keywords: *Biomaterials, epoxidized palm oil, epoxidized rbo, vegetable oils, ATR-FTIR.*

### Introduction

Vegetable oils are predominantly with fatty acids esterified to triacylglycerides (TAG), which mainly consist of unsaturated C16 – C22 fatty acids (Fomitšov, 2019). Vegetable oils,

especially palm olein, is widely used in Malaysia as edible oil for cooking and contain high saturated fat with low trans fats (Imosis *et al.*, 2015). Figure 1 shows the general triglyceride structure of common vegetable oil containing unsaturated bonds.

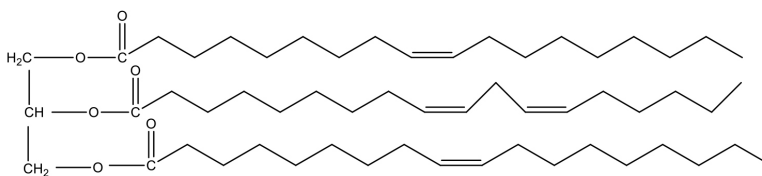


Figure 1: Chemical structure of lipid of vegetable oil

Vegetable oils can be extracted from the seeds and the fruits of the plants. Soybean oil and sunflower oil are the examples of oil that are extracted from the seeds while palm oil, olive oil and rice bran oil are the examples of oil extracted from the fruit of the plant (Bulley *et al.*, 1984). Vegetable oil is widely used to substitute fossil fuel as feedstock because of its stable chemical

and physical properties (Fomitšov, 2019; Kouzu & Hidaka, 2012; Nyashina & Strizhak, 2019). Vegetable oil is a renewable source with great properties such as high flash point, high lubricity, biodegradable and non-toxic (Borugadda *et al.*, 2017). The physical properties of vegetable oils show that they can be used to synthesize a new biomaterial as they are safe to the environment.

Table 1 shows typical fatty acid compositions in the edible vegetable oils as expressed by percentage weight of fatty acid methyl ester by gas chromatography analysis.

Table 1: Fatty acid composition in selected edible oils

FAs (%)	Palm Oil <sup>1</sup>	Rice Bran Oil <sup>2</sup>	Canola Oil <sup>3</sup>	Soybean Oil <sup>4</sup>	Sunflower Oil <sup>2</sup>
C12:0	0.2	nd	nd	nd	0.02
C14:0	1.1	0.39	nd	nd	0.09
C16:0	44.0	20.0	4.3	11.5	6.2
C16:1	nd	0.19	0.2	nd	0.12
C17:0	nd	nd	nd	nd	0.02
C18:0	4.5	2.1	1.8	3.62	2.8
C18:1	39.2	42.7	62.3	24.0	28.0
C18:2	10.1	33.1	19.4	54.6	62.2
C18:3	0.4	0.45	9.2	5.87	0.16
C20:0	0.1	nd	0.60	0.46	0.21
C20:1	nd	1.11	1.20	Nd	0.18
C22:0	nd	nd	nd	Nd	nd

nd, not detected; <sup>1</sup>Mancini *et al.* (2015); <sup>2</sup>Orsanova *et al.* (2015); <sup>3</sup>Ghazani *et al.* (2014); <sup>4</sup>Sahoo *et al.* (2015)

From Table 1, the compositions of fatty acids in each oil are varied according to saturation and unsaturation moieties of the fatty chains. As such, palm olein consists of 49.9% saturated fatty acids (SFAs), followed by 39.2% monounsaturated fatty acids (MUFAs) and only 10.5% polyunsaturated fatty acids (PUFAs). Meanwhile, rice bran oil contains higher number of MUFAs (44.0%) together with PUFAs (33.5%) and almost 22.5% of SFAs. The MUFAs are the most abundant fatty acids in canola oil, amounting to 63.7%, followed by 26.6% PUFAs and only 6.7% SFAs. The fatty acid compositions of soybean and sunflower oils somewhat fall in the range of 9.34-15.58% of SFAs, MUFAs 24.0%-28.3% and 60.47%-62.36% PUFAs.

Due to their versatility and structure, vegetable oils can be modified by few chemical reactions into other products such as esterification, epoxidation, hydrolysis, saponification and transesterification reactions, whereas the epoxidation process will produce epoxidized oils. This reaction involves the

addition of single oxygen atom into either monounsaturated or polyunsaturated carbon-carbon double bonds in the fatty acid chains by percarboxylic acids or organic/inorganic peroxides as oxidizing agent (Omonov *et al.*, 2016). This will eventually change the original chemical structure into epoxide, or also known as oxirane or epoxy group, with a structure of cyclic with three membered ring structures. Epoxide has highly strained ring structure which makes the epoxy group more reactive than other ethers. This makes epoxy group important as intermediates for a variety of products (Arniza *et al.*, 2015; Goud *et al.*, 2006).

During the epoxidation process, the high content of unsaturated carbon-carbon double bond is more desirable. Thus, the fatty acid constituent of vegetable oils can be used to assume the content of the epoxy group formed (Pérez *et al.*, 2009). The epoxy group that forms in the vegetable oil can be monitored directly with oxirane oxygen content analysis and also by iodine value analysis for indirectly monitoring (Parreira *et al.*, 2002). The purpose of this paper

is mainly to show how vegetable oils could be modified by simple epoxidation reaction to further serve as renewable material feedstock. While fatty acid profiles of commodities such as sunflower, soy bean and canola oils are major lipid of interest in the world (Starostina *et al.*, 2020), local commodity in Malaysia such as palm oil was also given consideration in this study.

## Materials and Methods

### Chemicals and Materials

The starting oils of palm olein, rice bran oil, canola oil, soybean oil and sunflower oil were all purchased from the local supermarket. Additionally, chemicals and reagents such as formic acid (reagent grade 95%), hydrogen peroxide, sodium chloride, sodium hydroxide and n-hexane were attained from Sigma-Aldrich (Malaysia).

### Epoxidation Reaction of Edible Oils

The epoxidation of edible oils was carried out according to the previous report by Omanov *et al.* (2016) with slight modifications. Each of the edible vegetable oil (25 g) was weighed using analytical weighing balance. The epoxidation was carried out by adding 23 g of formic acid (reagent grade 95%) and 102 g of 30% hydrogen peroxide to each of the starting oil with the ratio of 1:1:6 (edible oil: formic acid: hydrogen peroxide, w/w). In brief, formic acid and vegetable oil were mixed together in a 100 mL beaker and the mixture solution was then poured into the three neck flasks of a reflux system. Hydrogen peroxide was added drop wise to the solution mixture, and this process

was completed in one hour with the temperature of the mixture maintained at (45 to 50°C). The mixture was stirred continuously at 10 rpm speed to ensure its homogeneity. The reaction was monitored closely since the exothermic reaction has occurred due to reactive reaction between formic acid and hydrogen peroxide. The reaction temperature was then increased to 60 to 65°C for another 6 hours. After 6 hours, the epoxidized oil was cooled at room temperature before continuing with the next process. All of the processes were carried out in the fume hood.

### Liquid-Liquid Extraction of Epoxidized Oils

Liquid – liquid extraction (LLE) is a method to separate two immiscible solutions, in which solvent polarity index was used to identify the suitable solvent to separate between two immiscible layers. Approximately, 10 g of epoxidized oil samples was weighed. Meanwhile, 5 g of sodium hydroxide (NaOH) and sodium chloride (NaCl) were separately weighed and then diluted with distilled water up to the scale of the volumetric flasks (100 mL). The oil samples were poured into the separating funnel followed by 15 mL of NaOH solution, 15 mL of NaCl solution and 15 mL of n-hexane. The schematic Figure 2 shows the layers of the mixture due to the differences in density and polarity of the solutions and samples. The function of NaOH in this study was to neutralize formic acid (strong acid) that might be residually present in the oil samples. While, the function of NaCl was to absorb the left-over moisture that is in the oil samples (Mungroo *et al.*, 2008). Besides, the function of n-hexane was to break the emulsion between formic acid and hydrogen peroxide that still remained in the epoxidized oil samples.

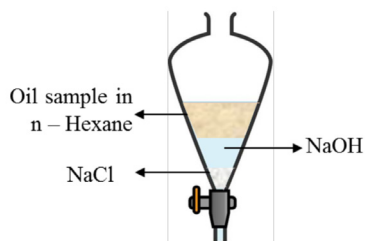


Figure 2: The layer of the epoxidized oil in LLE solvent mixtures

### **Chemical Characterization Using Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy**

In this study, the chemical analysis for the vegetable oils as starting material (not shown in the results) and epoxidized oils were carried out using a Shimadzu Fourier Transform Infrared spectroscopy with Attenuated Total Reflectance mode (ATR-FTIR, Shimadzu, Japan) in the mid-infrared wavelength of  $4000 - 400 \text{ cm}^{-1}$ . In brief,  $10 \mu\text{L}$  of oil samples was dropped onto the ATR diamond accessory and each sample was analyzed with  $4 \text{ cm}^{-1}$  resolution and at 40 scans each. The spectrum obtained from the analysis underwent manipulation process such as

baseline correction, normalization, smoothing and peak assignment in order to increase the image resolution and visual properties.

### **Results and Discussion**

A complete modification of palm olein, rice bran oil, canola oil, soybean oil and sunflower oil with the presence of reagents ( $\text{CH}_2\text{O}_2$  and  $\text{H}_2\text{O}_2$ ) has produced epoxidized oils. The formation of epoxy group occurred when the solvent reacted with carbon-carbon double bond in the chemical structure (Chandrasekara *et al.*, 2011). Figure 3 shows the mechanism example of the conversion of palm olein into epoxidized palm oil.

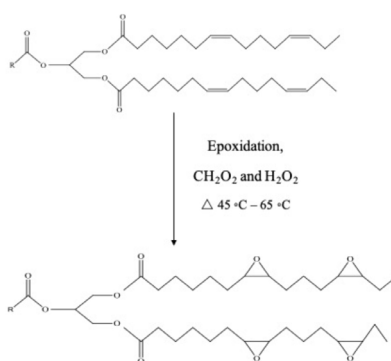


Figure 3: Mechanism of the synthesis of epoxidized palm oil

The oxirane ring in the structure was formed by the addition of nucleophile that was present in the  $\text{CH}_2\text{O}_2$  and the homogenous acid catalyst  $\text{H}_2\text{O}_2$  (Centi *et al.*, 2000; Nor *et al.*, 2017). In the reaction, the nucleophilic addition was more favorable due to the number of carbon-carbon

double bond and no other reaction occurred during the process. In this study, the physical characteristic that can be observed by naked eyes was the texture of the oil, as illustrated in Figure 4.

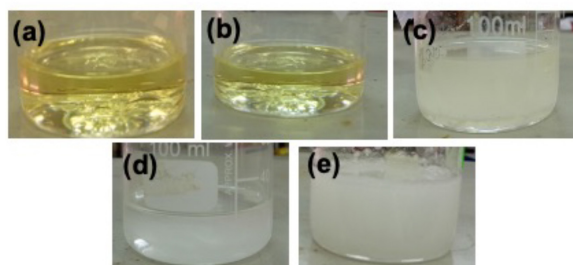


Figure 4: Variation of textures of epoxidized oil samples: (a) palm oil, (b) rice bran oil, (c) canola oil, (d) soybean oil and (e) sunflower oil

Initially, the texture of each starting oil form was in the same liquid state and yellowish in color, however after the modification, the canola oil, soybean oil and sunflower oil visually became clearer and lighter in colour. These changes were in agreement with the previous findings by Sienkiewicz and Czub (2016) where they found that the epoxidation of soybean process oil resulted in a formation of clearer and yellowish liquid oil, regardless of the reaction conditions. The colour changes were mainly due to the SFAs fatty acid composition in oils, where the percentage of SFAs in these three respective oils is lower than others (Table 1).

FTIR spectra indicated the functional groups that were presented in the chemical

structure. This technique detects the vibration of a molecule in the compound, thus providing the various types of bonds that exist (Boyatzis *et al.*, 2016). In this work, each of the oils showed discrepancies in peak range of oxirane ring functionality due to the variation of numbers of unsaturated carbon-carbon double bond that was present in the oil structure. Moreover, the intensity of the peaks (strong, medium, and weak) showed whether the formation of oxirane ring was abundant or not. Table 2 shows the assignment for most characteristic Infrared band of palm olein, rice bran oil, canola oil soybean oil and sunflower oil that was previously reported (Hashim *et al.*, 2017; Irnawati *et al.*, 2020; Purbaya *et al.*, 2015; Setyaningrum *et al.*, 2013; Shi *et al.*, 2017).

Table 2: ATR-FTIR absorption characteristics of vegetable oils based on reference studies

Functional Group	Wavenumber (cm <sup>-1</sup> )				
	Palm Oil <sup>1</sup>	Rice Bran Oil <sup>2</sup>	Canola Oil <sup>3</sup>	Soybean Oil <sup>4</sup>	Sunflower Oil <sup>5</sup>
C-H str (next to C=C) <i>cis</i>	3008	3007	3008	3005	3008
C-H asymmetry str	2953, 2922	2953, 2922	2958, 2925	2953, 2922	2925
C-H symmetry str	2853	2853	2854	2852	2854
C=O str	1744	1743	1747	1746	1746
C=C str	n.m	1654	n.m	1654	n.m
C-C str	n.m	1237	n.m	n.m	1235
C-O str	1160	1159-1096	n.m	1160	1163
C-H <sub>3</sub> , CH <sub>2</sub> bend	1461	1460, 1378	1465, 1376	1463, 1377	1464, 1377

n.m: not mentioned; <sup>1</sup>Hashim *et al.* (2017); <sup>2</sup>Irnawati *et al.* (2020); <sup>3</sup>Shi *et al.* (2017); <sup>4</sup>Setyaningrum *et al.* (2013); <sup>5</sup>Purbaya *et al.* (2015)

The data showed a typical characteristic of absorption peaks for common triglycerides in edible oils. Based on the previous studies, absorption bands at  $n=3008 - 3005 \text{ cm}^{-1}$  are notable for the stretching vibration for the *cis* double bonds of unsaturated fatty acids. However, in the present study, this unsaturated C-H stretching peak of hydrocarbon chain would later disappear from the spectra of epoxidized oils (see Table 3), with the exception for the epoxidized soybean oil. This indicated that the unsaturated fatty acid moieties of the edible oils have turned into epoxide. Additionally, in pure unmodified oils, strong band absorptions were usually observed in the region of  $n=2953 - 2852 \text{ cm}^{-1}$  which were attributed to C-H asymmetry stretching vibrations. The bending vibrations of methyl ( $\text{CH}_3$ ) and methylene ( $\text{CH}_2$ ) groups are also reported in the range of  $n=1465 - 1460 \text{ cm}^{-1}$  and  $n=1378 - 1376 \text{ cm}^{-1}$ . Previous studies also reported the appearance of large peak at  $n=1747 - 1743 \text{ cm}^{-1}$  which corresponded to C=O double bond stretching vibration in

triglyceride of all selected oils, together with stretching vibration peak of C-O in the region of  $n=1163 - 1096 \text{ cm}^{-1}$  (Hashim *et al.*, 2017; Irnawati *et al.*, 2020; Purbaya *et al.*, 2015; Setyaningrum *et al.*, 2013; Shi *et al.*, 2017).

The ATR-FTIR absorption characteristics for palm olein, rice bran oil, canola oil soybean oil and sunflower oil are shown in Figures 5 to 9. In the FTIR spectrum of epoxidized palm olein (Figure 5), the weak intensity of  $\text{CH}_2\text{-O-CH}$  stretching was present at  $n=1250 \text{ cm}^{-1}$  and bending at  $n=1098 \text{ cm}^{-1}$ , together with oxirane peaks at range of  $n=1462 \text{ cm}^{-1}$  and  $900 - 800 \text{ cm}^{-1}$ . This intensity indicated that the quantity of oxirane ring present in the chemical structure was low. This spectrum was compared to epoxide palm oil acrylate from other analysis of Salih *et al.* (2015). The comparison between these two epoxide oils proved that formation of epoxide from palm oil product with the presence of C-O-C functional group at the fingerprint region.

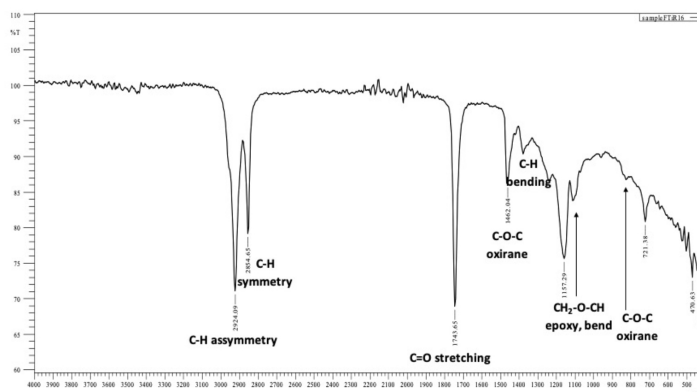


Figure 5: ATR-FTIR spectrum of epoxidized palm oil

Previously, Tavassoli-Kafrani *et al.* (2017) successfully used ATR-FTIR method to determine the oxirane oxygen content (OOC) produced in the epoxidation process of vegetable oils by measuring the changes in epoxy functional group absorption in the range of  $n=1497 - 1432 \text{ cm}^{-1}$ . In this current work, peaks at  $721.38 \text{ cm}^{-1}$  and  $820 \text{ cm}^{-1}$ , albeit low intensity, were attributed to the presence of the

C-O-C oxirane, and in agreement with previous findings reported by Vlcek and Patrovic (2006). Additionally, the methyl group ( $\text{CH}_3$ ) in the chemical structure showed strong intensity at  $n=2924.09 \text{ cm}^{-1}$  C-H asymmetry stretching, while methylene ( $\text{CH}_2$ ) showed strong intensity at  $n=2854.65 \text{ cm}^{-1}$  C-H symmetry stretching, and the C-H in deformation in the figure at  $n=1377 \text{ cm}^{-1}$ , all showing the back bone of fatty

acid chain of epoxide products. The fatty acid structure in palm oil with a balance of saturated and unsaturated constituents hence contributed to the lower oxirane formation, in comparison to other types of oils (Chandrasekara *et al.*, 2011; Salih *et al.*, 2015).

The functional groups for epoxidized rice bran and canola oils were shown in FTIR spectra in Figures 6 and 7 and tabulated in Table 3. In FTIR spectrum, the C-O-C epoxy

functionality was attributed to the peaks at  $n=1462\text{ cm}^{-1}$ ,  $820\text{ cm}^{-1}$  and  $721\text{ cm}^{-1}$ , which indicated the formation of oxirane groups in both oils. Similarly, additional peaks of  $\text{CH}_2\text{-O-CH}$  stretching and bending were present in the C-O region of  $n=1247\text{ cm}^{-1}$  and  $1107\text{ cm}^{-1}$ , similar to the recent findings reported by Tran *et al.* (2020) and Nicolice *et al.* (2010). On the other hand, the methyl group ( $\text{CH}_3$ ) showed strong intensity at  $\sim n=2924\text{ cm}^{-1}$ ,  $\text{CH}_2$  at  $n=2854\text{ cm}^{-1}$  and C-H bending at  $n=1377\text{ cm}^{-1}$ .

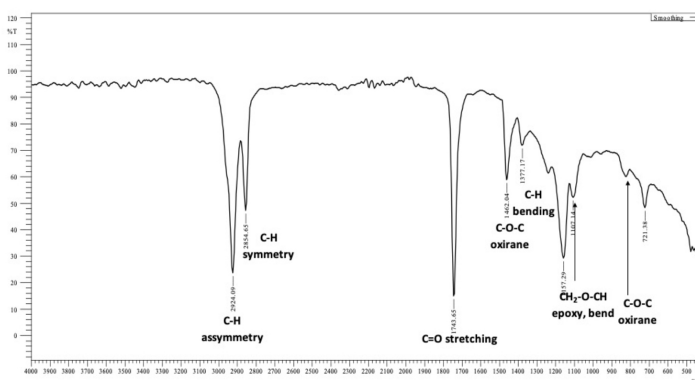


Figure 6: ATR-FTIR spectrum of epoxidized rice bran oil

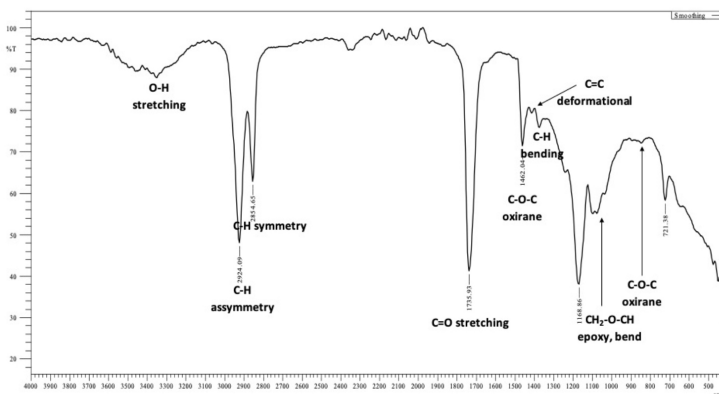


Figure 7: ATR-FTIR spectrum of epoxidized canola oil

The difference between epoxidized palm oil and rice bran oil with canola oil is that the canola oil reaction products showed another reading in the range of  $n=1420\text{ cm}^{-1}$ , possibly from  $\text{-C=CH}$  deformational, a characteristic peak for unsaturated bond, and comparative

to the same peak reported by Sienkiewicz and Czub (2016) at  $n=1418\text{ cm}^{-1}$ . This suggested that the oils contained unmodified double bonds, but relatively in small amounts since the intensity of the peak was small. In the FTIR spectrum of epoxidized canola oil, the formation of O-H

stretching at  $n=3360\text{ cm}^{-1}$ , possibly indicated the presence of moisture in the sample. Both rice bran and canola oils can undergo a variety of processes that can change their chemical structure into epoxide. The common process that has been used is transforming unsaturated carbon double bond into oxirane ring, by breaking the bond by adding performic acid as the main oxidizing agent (Borugadda *et al.*, 2017). These oils have lower polyunsaturated carbon double bond compared to soybean oil and sunflower oil, and also have high monounsaturated carbon double bond. Due to this, the oils have a high ability to change the chemical structure into epoxy group effectively (Adebiyi *et al.*, 2008; Przybylski, 2005).

Figures 8 and 9 represent the ATR-FTIR spectra of soybean oil and sunflower oil after epoxidation reaction. Both spectra show the presence of vibrational band at  $n=3400 - 3300\text{ cm}^{-1}$  for O-H stretching with weak intensity, was attributed to the presence of moisture in the oil samples. The C-O-C oxirane functional group for epoxide functionality were consistently shown at  $n=1462\text{ cm}^{-1}$  with strong intensity and  $n=820\text{ cm}^{-1}$  at weak intensity. Additionally, the presence of strong absorption at  $n=1095\text{ cm}^{-1}$  was attributed to the presence of  $\text{CH}_2\text{-O-CH}$  epoxy bending, somewhat similar to the report by Nikolic *et al.* (2010), although this peak was slightly shifted to the left in this work. This indicated that there was a high amount of oxirane ring form in both oils.

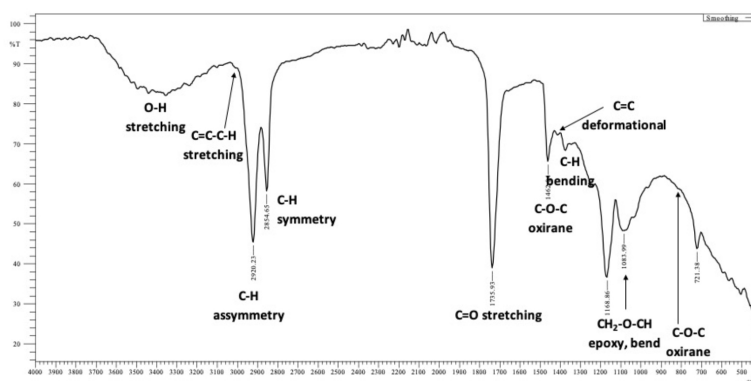


Figure 8: ATR-FTIR spectrum of epoxidized soybean oil

However, the presence of  $-\text{C}=\text{CH}$  deformational characteristic peaks ( $\sim n=1420\text{ cm}^{-1}$ ) in both oil samples and additional band attributed to the C-H stretching next to carbon-carbon double bond,  $-\text{C}=\text{C}-\text{H}$  ( $n=3010\text{ cm}^{-1}$ ) in epoxidized soybean oil also suggested the oils still contained the unmodified double bonds. Previously, the presence of low intensity peak at the range of  $n=3017 - 3004\text{ cm}^{-1}$  was used to measure the relative changes in iodine value (represent carbon-carbon double bond)

of unreacted edible oils (Tavassoli-Kafrani *et al.*, 2017). Basically, the process in changing the chemical structure of these types of oils into epoxy functional group is the same for the other vegetable oils. However, since their TAG oil structures have relatively high PUFAs carbon double bonds ( $>60\%$ ), refer to Table 1) as compared to monounsaturated carbon double bonds, these oils have low ability to change all the unsaturated carbon double bond into epoxy group (Adebiyi *et al.*, 2008; Adhvaryu & Erhan, 2002).



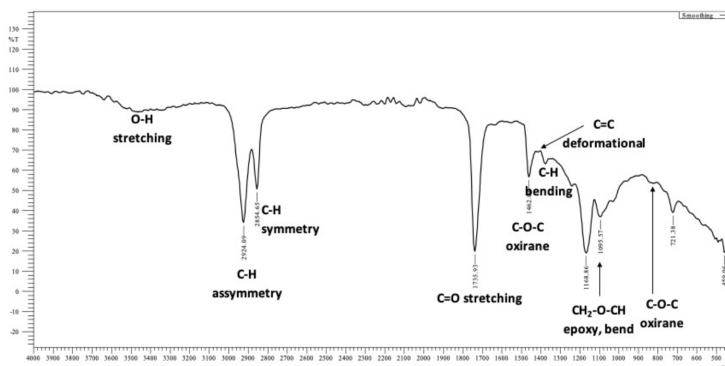


Figure 9: ATR-FTIR spectrum of epoxidized sunflower oil

Table 3 tabulates the FTIR peaks assignment that were present in the ATR-FTIR spectra in Figures 5 to 9. It is worth noting that in order to ensure all the edible oils turn into epoxidized oils, the reaction process must be consistent

and precise. These included the weight of the oil taken, ratio of oil and solvent, temperature used, volume of solvent and chemical analysis. The process should be consistent and precise to significantly archive high formation of oxirane ring (Mungroo *et al.*, 2011).

Table 3: Respective ATR-FTIR absorption bands of the epoxidized edible oils

Functional Group	Wavelength Peak (cm <sup>-1</sup> )				
	Epoxidized Palm Oil	Epoxidized Rice Bran Oil	Epoxidized Canola Oil	Epoxidized Soybean Oil	Epoxidized Sunflower Oil
O-H str	-	-	3360	3300	3400
C-H str (next to C=C) <i>cis</i>	-	-	-	3010.25	-
C-H asymmetry str	2924.09	2924.09	2924.09	2920.65	2924.09
C-H symmetry str	2854.65	2854.65	2854.65	2854.65	2854.65
C=O str	1743.65	1743.65	1735.93	1735.98	1735.93
C-C oxirane	1462.04	1462.04	1462.04	1462.04	1462.04
C-O-C oxirane	836.10	841.11	850.35	825.03	820.16
C-H bend	1377.10	1377.17	1377.14	1377.14	1377.12
CH <sub>2</sub> -O-CH epoxy, bend	1080.79	1107.14	1050.13	1083.99	1095.57
C=C deformation	-	-	1420.86	1420.86	1420.86

**Conclusion**

In conclusion, edible oils could have great potential as starting materials in the future synthesis of biobased epoxy products, owing to their high availability, versatility and fatty

acid compositions. Modification process such as epoxidation under mild reaction condition will produce biobased feedstock with great properties such as low toxicity and desirable oxidative stability. During the epoxidation process of five different edible oils with

different fatty acid constituents, it revealed that the starting oils with high unsaturated double bond content are more preferable because of higher ability for oil transformation into epoxide. Additionally, ATR-FTIR technique can be used as a rapid measurement and simple screening for the determination of epoxidation process of oil. However, this method can be further optimized for quantitative measurement such as in the calculation of percentage functional group conversion for the oxirane oxygen content. Additionally, other advanced chromatography techniques such as HPLC-MS and H-NMR spectroscopy should also be considered as characterization methods. The obtained epoxidized oils can be further used in the ring opening reactions and polymerization for new biobased materials.

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