



Synthesis and Physicochemical Properties of Epoxidized Rubber Seed Oil from Acetic and Formic Acids under Identical Reaction Conditions

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ABSTRACT: The main objective of this article was to report the synthesis and the physicochemical properties of epoxidized rubber seed oil (ERSO) from acetic and formic acids under identical reaction conditions at 60 °C. The effect of organic acids on epoxy content and other physicochemical properties were determined. The differences observed in the ERSO samples were explained in terms of the structural parameters derived from FTIR, ¹H, and ¹³C NMR spectroscopy. The physicochemical characteristics of the ERSO samples were similar to those of the currently available commercial epoxy vegetable oils.

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Epoxidized oils are of extreme commercial value because of many reactions they undergo. Vegetable oils such as coconut oil, linseed oil, olive oil, palm oil, rapeseed oil, soybean oil, and sunflower oil have been explored for the production of epoxides (Mohammed *et al.*, 2020). These oils are predominantly composed of triglyceride molecules with varied fatty acid chains. The physicochemical properties of vegetable oil triglyceride molecules are dependent on the length of fatty acyl chains, number and type of unsaturation in the chains, and distribution of fatty acyl groups present in the Sn-positions of the triglycerides (Fei *et al.*, 2018). Epoxidation of fatty acids/triglycerides is the reaction of a carbon-carbon double bond with active oxygen, which results in the addition of an oxygen atom,

converting the original double bond into a three-membered epoxide (oxirane) ring (Moser *et al.*, 2022). Epoxidation is commonly carried out using a peracid (peroxy acid), either preformed or formed *in situ*, by reacting carboxylic acid as an oxygen carrier to the double bonds with hydrogen peroxide (H₂O₂) as an oxygen donor (Okieimen *et al.*, 2002; Saraswat *et al.*, 2024). The *in situ* epoxidation of vegetable oil is a well-known procedure for epoxidation carried out on a laboratory scale. Similarly, this method has been scaled up on a large scale through batch reaction production (Cooney *et al.*, 2011). Formic acid and acetic acid are commonly used as carboxylic acids for *in situ* epoxidation of vegetable oils, as reviewed in previous studies (Moser *et al.*, 2022; Saraswat *et al.*, 2024). Thus, the epoxidation of vegetable oils

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using an *in situ* method to obtain the optimal epoxide content and high transformation selectivity are determined by temperature, peracid–olefinic unsaturation molar ratio, reaction time, amount of catalyst, and stirring speed (Gan *et al.*, 1992; Okieimen *et al.*, 2002). Although, the yield of epoxide content may be lowered due to possibility of a competing hydroxylation reaction with the resultant opening of the epoxide ring (Okieimen *et al.*, 2002; Okieimen *et al.*, 2005). This situation is further complicated by the fact that the conditions that favour the yield of epoxide, particularly the use of mineral acid as a catalyst, slightly higher reaction temperature than normal, and the molar ratios of the reactants, also favour the competing hydroxylation reaction (Rangarajan *et al.*, 1995). Dinda and colleagues found that the rate of formation of epoxide was higher with formic acid than with acetic acid (Dinda *et al.*, 2008). Although acetic acid was later found by other workers to increase ethylenic unsaturation conversion to epoxides by 10% while reducing the formation of unwanted by products (Cooney *et al.*, 2011; Mungroo *et al.*, 2008). This observation was previously attributed to the relatively higher activity of the formic acid, resulting in the rapid decomposition of hydrogen peroxide, thus leaving the reaction batch depleted of oxygen (Petrović *et al.*, 2002). In our previous studies, we investigated the kinetics of epoxidation of RSO with acetic acid and formic acid, and estimated the values of the rate constant for epoxidation of RSO at different temperatures on the order of $10^{-6} \text{ L mol}^{-1} \text{ s}^{-1}$ with both acids. The E_a for epoxidation of RSO are respectively $15.73 \text{ kcal mol}^{-1}$ and $65.81 \text{ kcal mol}^{-1}$ for acetic and formic acid, respectively (Okieimen *et al.*, 2002). The average values of the thermodynamic parameters were found to be $S = -83.2 \text{ J mol K}^{-1}$ and $G = 36.2 \text{ KJ mol}^{-1}$ for acetic acid compared with $S = -84.8 \text{ J mol K}^{-1}$ and $G = 26.7 \text{ KJ mol}^{-1}$ obtained for formic acid. Despite a reasonable number of published works on epoxidized rubber seed oil from both acetic acid and formic acid, the final products have not been fully characterized and compared with commercially available epoxides. Epoxidized RSO and its metallic soaps have been used as plasticizers / stabilizers for PVC (Balköse *et al.*, 2008). The epoxidation of vegetable oils improves their stability and provides adequate reactivity to form chemical linkages with other polymer chains. More recently, Aigbodion *et al* reported the application of RSO and epoxidized RSO as natural rubber additives (Aigbodion *et al.*, 2000). They suggested that ERSO with higher epoxy content may yield better results as a modifier of natural rubber. Considering the foregoing facts, the present work was carried out to synthesis and determined the physicochemical

properties of epoxidized rubber seed oil (ERSO) from acetic and formic acids under identical reaction conditions.

MATERIALS AND METHODS

Materials: Rubber seeds were collected from the 40-ha Estate of the Rubber Research Institute of Nigeria, Benin City. The 15 years old estate was planted with NIG 800 series RRIN developed high-latex-yielding clones. RSO was obtained by solvent extraction from the seed using n-hexane (boiling point 60°C). The physico-chemical properties of the oil were determined by AOCS method (AOCS, 1998). Formic acid (98%), acetic acid (99.5%), H_2O_2 (30%) and HBr were obtained from Merck. All the chemicals and reagents used in this study were of analytical grade and purchased from Sigma-Aldrich, Germany, and used as received.

Synthesis of epoxidized rubber seed oil: ERSO was synthesized from rubber seed oil using peroxy acetic acid or performic acid prepared *in situ* by reacting acetic acid or formic acid with hydrogen peroxide in the presence of small quantities of 5mmol of concentrated sulphuric acid. In a typical experiment, glacial acetic acid (5.0g, 0.084 mol) and RSO (40.0g, 0.0482mol) were charged into a 250-mL reaction flask and the reaction mixture was maintained at 10°C . 30% hydrogen peroxide (46.1g, 0.407mol) equilibrated at 10°C , was added drop wise under continuous stirring for 1h. Thereafter, the reactor temperature was raised and maintained at 60°C for 8h under magnetic stirring. The aliquots of the reaction mixture were cooled to 40°C temperature. The oil was extracted with ethyl acetate, separated and washed with a brine solution (5% wt NaCl) until acid free and dried over sodium sulphate. The epoxidized oil was concentrated by removing the solvent in vacuo at 60°C . The resulting ERSO was characterized using standard method (AOCS, 1998). Another reaction was repeated for 100% formic acid (4.9g, 0.106mol), RSO (40.0g, 0.0482mol) and 30% hydrogen peroxide (46.1g, 0.407mol). H_2SO_4 was not used as catalyst in this epoxidation with formic acid.

The relative conversion to oxirane (R_{CO}) was calculated as follow (Gunawan *et al.*, 2023)

$$R_{CO} = \left(\frac{R_{Oex}}{R_{Oth}} \right) \times 100 \quad (1)$$

where O_{Oex} is the content of oxirane oxygen experimentally determined, and O_{Oth} is the theoretical maximum oxirane oxygen in 100 g of RSO oil obtained from the expression (Gunawan *et al.*, 2023).

$$O_{oth} = \frac{\left(\frac{IV_0}{2A_i}\right)}{\left[100 + \left(\frac{IV_0}{2A_i}\right)A_o\right]} \times A_o \times 100 \quad (2)$$

Where A_i (126.9) and A_o (16.0) are the atomic weights of iodine and oxygen, respectively; and IV_0 is the initial iodine value of RSO.

Spectroscopy Characterization of RSO and ERSOs: The chemical structures of RSO and ERSO were characterized by nuclear magnetic resonance (NMR) and Fourier transform infrared (FTIR) and spectroscopy. The ^1H - and ^{13}C -NMR characterization of RSO and ERSOs were analyzed with Bruker 300-MHz NMR spectrophotometer (Bruker Instruments Inc., Karlsruhe, Germany). CDCl_3 containing a small amount of tetra methyl silane as an internal standard was used as solvent. Infrared characterizations were analyzed with an impact 400 D Nicolet FTIR spectrophotometer (Madison, WI). The samples were spread over NaCl cells and the Fourier transform infrared spectrometer scanned 32 times in the measurement range $4000\text{--}400\text{ cm}^{-1}$.

RESULTS AND DISCUSSION

Chemical characterization of RSO: The kernel of the rubber seed has an oil content of 46.12 %. The composition of RSO determined by GC-MS revealed the presence of triglycerides containing one, two, and three unsaturated fatty acids, as discussed in our previous study (Bakare *et al.*, 2008). Rubber seed oil contains unsaturated fatty acids comprising of 20.0% oleic acids, 36.0% linoleic acid, and 23.5% linolenic acid. The saturated fatty acids present are mainly myristic acid (22.0), palmitic acid (7.6%) and stearic acid (10.7%). The iodine value of rubber seed oil (Table 1) in comparison to its fatty acid further corroborates its level of unsaturation. The iodine value of 134.5 g $\text{I}_2/100\text{ g}$ recorded for RSO in this study falls within the range of semi-drying oil, which

is a potential raw material in the paints, resins, polyols, and lubricating oil industries.

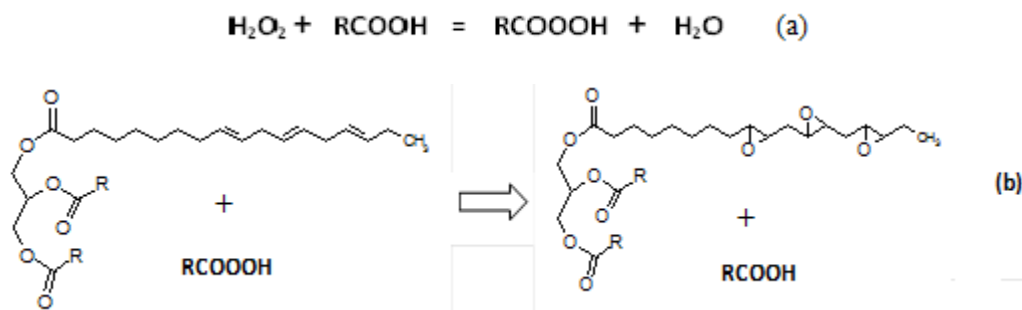
Table 1: Physico-chemical properties of rubber seed oil (RSO)

Properties	RSO
Specific gravity (30°C)	0.916
Acid value (mg KOH/g)	42.49
Free fatty acid (% as oleic acid)	20.84
Saponification value (mg KOH/g)	201.4
Iodine value (g $\text{I}_2/100\text{g}$)	134.5

The double bonds contained in this oil can be converted into more reactive epoxy groups through epoxidation reactions. The seed oils currently employed in the commercial production of epoxy esters (Shaah *et al.*, 2021) share similar properties with RSO results shown in Table 1.

The synthesis of epoxidized rubber seed oil: The synthesis of epoxidized rubber seed oil is outlined in two steps reaction processes in scheme 1. The reversible reaction of formic/acetic acid with hydrogen peroxide to form peroxyacid (Scheme 1a) has been well established as the rate limiting phase (Okieimen *et al.*, 2002; Rangarajan *et al.*, 1995). The peroxyacid generated in the aqueous phase is immediately used up in the oil phase of the epoxidation (Scheme 1b) process. Although, the rate of formation of peracetic acid is slower than the rate of performic acid production, consequently H_2SO_4 was added as catalyst to speed up the rate of peracetic acid formation (Rangarajan *et al.*, 1995).

Fourier Transform Infrared Spectroscopy (FTIR) studies: The spectroscopic properties of RSO, ERSO-Ac, and ERSO-Fo were studied and compared using Fourier transform infrared (FTIR) spectroscopy. The FTIR spectra of the RSO and its derivatives are shown in Figure 1. The RSO spectrum presents the characteristic absorption bands of common triglycerides (Guillén *et al.*, 2004; Okieimen *et al.*, 2005). The FTIR evaluation of the RSO spectrum has been fully discussed in our previous reports (Bakare *et al.*, 2008; Okieimen *et al.*, 2005).



Scheme 1: The synthesis of epoxidized rubber seed oil: Formation of (a)-peroxyacid and (b)- epoxide

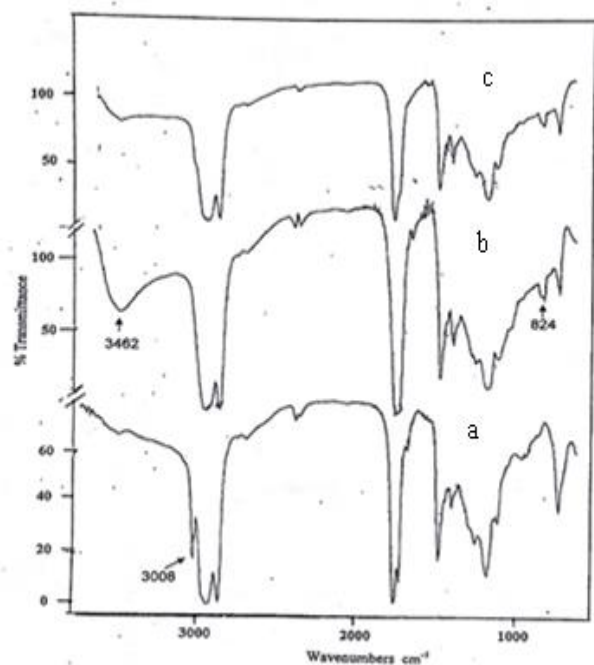


Fig. 1: FTIR spectra of (a) RSO, (b) ERSO-Ac, and (c) ERSO-Fo

The characteristic band of special interest in this study includes the C-H stretching vibration of olefinic double bonds near 3008 cm^{-1} ; the two methylene asymmetrical and symmetrical stretching vibrations at approximately 2925 and 2854 cm^{-1} respectively; a small band which appear as sharp shoulder at 1654 cm^{-1} assigned to C=C of unsaturated acyl groups; the sharp band at 1419 cm^{-1} resulting from the bending vibrations of the CH_2 and CH_3 aliphatic groups; and the sharp band at 723 cm^{-1} due to the rocking vibrations of $-(\text{CH}_2)_n-$ bonds of olefins. Changes in infrared spectrum of the RSO oil after epoxidation reaction showed changes in the resulting spectra of epoxidized rubber seed oil by acetic acid and formic acid (Figure 1b and c). The two most interesting spectral regions where (i) the vibrational activity of non-conjugated bonds of olefinic and the bending vibrations of aliphatic compounds which were observed at 700 to 1500 cm^{-1} , and (ii) the stretching vibrations of the acyl chain region at $2800 - 3800\text{ cm}^{-1}$ were observed. Both ERSO-Ac and ERSO-Fo spectra displayed a sharp band in the fingerprint region at 824 cm^{-1} attributed to epoxy ring. The disappearance of the double bond absorption bands at 1654 cm^{-1} in ERSO-Ac and ERSO-Fo further corroborates the conversion of the olefinic acyl chain to the epoxy group. The stretching vibration at 3008 cm^{-1} of the olefinic CH double bonds completely disappears in both epoxy spectra.

However, there was an additional broad band at 3400 cm^{-1} due to the free hydroxyl group resulting from the epoxy ring opening in the ERSO-Ac spectrum. Adhvaryu *et al* (Adhvaryu *et al.*, 2005) earlier reported that H-bonding may results in broadening of the -OH absorption. Hence, the presence of intermolecular H-bond increases the viscosity of ERSO-Ac compared with ERSO-Fo.

The ^1H NMR spectra: The ^1H NMR spectrum of RSO oil gave nine signals of significant intensity. These signals are attributed to protons of the main components of the triglycerides acyl chain and are compared with signals of ERSO-Ac and ERSO-Fo (Figure 2). The area of these ^1H NMR spectra signals have been reported to be proportional to the number of protons of each type in the sample (Guillén and Ruiz, 2003).

Consequently, the degree of unsaturation and the proportions of the different acyl groups in the samples can be calculated from the area of some signals of the spectra (Guillén and Ruiz, 2003; Knothe and Kenar, 2004). The region where significant changes occur in the spectra are; the peaks of olefinic protons lying between $5.3 - 5.4$ ppm; protons attached to the *bis*-allylic carbons ($2.7 - 2.8$ ppm); and protons attached to the allylic carbons ($2.0 - 2.1$ ppm). The olefinic hydrogens (CH=CH) at $5.2 - 5.4$ ppm present in the ^1H NMR spectrum of RSO completely disappeared in ERSO-Ac and ERSO-Fo (Okieimen *et al.*, 2005).

This fact suggests that the C=C undergoes complete epoxidation. The peak of residual olefinic group (C=C) in the expanded spectra at 5.31 ppm has a small intensity and the allylic CH_2 hydrogen signal at $1.97 - 2.02$ ppm almost disappear. Rather, a new peak that centered at 3.06 ppm appeared in both ERSO-Ac and ERSO-Fo. Also, a variation was observed in the peak centering at 3.06 ppm and 2.7 ppm in both epoxy spectra compared with starting oil. The intensity of the peak centering at 3.06 ppm is higher and approximately 6 times compared with 2.7 ppm in epoxy acetic acid. However, reverse is the case with formic acid. It is worthy to note that no significant change occurred in the characteristic signal of spectra of glycerol proton which presents two signals at 4.29 and 4.15 ppm . This signals corresponded to 2H in both positions sn-1' and sn-3', respectively.

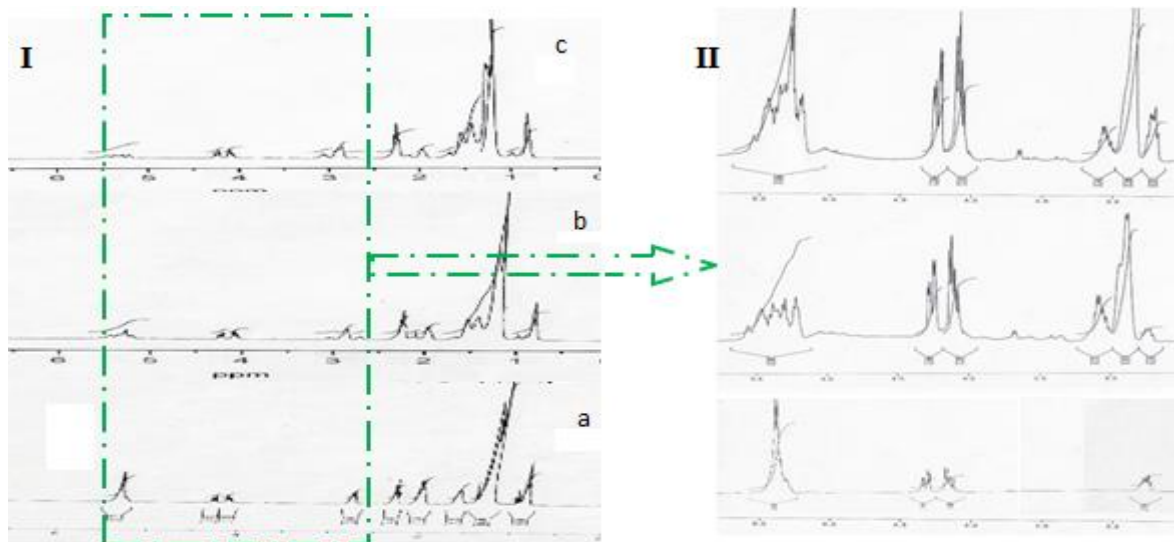


Fig. 2:(I) ^1H NMR spectra of (a) RSO, (b) ERSO-Ac, (c) ERSO-Fo and (II) Expanded spectra of ^1H NMR (a) RSO, (b) ERSO-Ac, and (c) ERSO-Fo

The ^{13}C NMR spectra: The ^{13}C spectra of RSO presented in Figure 3 showed the characteristic peaks of carbonyl, olefinic, glyceryl, methylene and saturated methyl group. These peaks are grouped into five distinct regions of important resonance peaks. Similarities between the ^{13}C NMR of RSO and its epoxidized derivatives were observed. Although, differences in the intensities of the peaks were observed in some specific region. The carbonyl carbon resonates between 173-172 ppm. The chemical shift at 173.3 ppm was attributed to the saturated carbon at 1/3 position. The signal at 173.3 and 172 ppm assigned to the oleate chain at the 1(3) and 2 positions, respectively. The olefin carbons are observed in the 130.3-127.5 ppm range. These olefin carbons are from mono- and poly unsaturation in the FA chain, which are mainly of oleic, linoleic and linolenic acyl groups of RSO. The differences in these signals are mainly due to their proximity to the ester group. It was observed that after epoxidation of rubber seed oil, the chemical shift of olefin carbons in Figure 3a decreased remarkably due to the formation of the epoxy group in Figures 3b and c. Although, the chemical shift of olefin carbons does not disappear completely due to residual effect. Since the chemical shift of the carbonyl carbon did not change during the epoxidation reaction, therefore, comparing the height of this carbonyl carbon with the olefinic carbon might give an insight into the reaction products. Conversely, it was observed the height of the four prominent olefinic carbon of RSO which appear between 131-127ppm was almost three times more than carbonyl carbon at centering at 173ppm.

However, these height intensities were about the same in both ERSO-Ac and ERSO-Fo. This observation is responsible for the iodine value of 12.5 and 18.11 g $\text{I}_2/100$ g of oil observed in ERSO-Ac and ERSO-Fo respectively (Table 2). The ^{13}C NMR of glycerol carbons detected at 62.4 and 68.8 ppm for C1/C3 and C2 respectively exhibited two signals. Interestingly, these signals increased to four major distinct signals (Figures 3b & c). The appearance of two additional new ^{13}C peaks at 53.9 – 56.9 ppm (Figs 3b & c) due to the epoxy carbon further confirmed the formation of the epoxide. The carbon resonances to the allylic (methylene carbon) and bis-allylic carbons of the olefinic, linolenic and linoleic acid appeared within the range of 34.1 and 20.2 ppm (Vlahov *et al.*, 2002). These clusters of methylene signals have been differentiated based on the proximity to the ester group (Vlahov *et al.*, 2002).

The Physicochemical properties of epoxidized rubber seed oil: The physicochemical properties of epoxidized rubber seed oil are presented in Table 2. These results are compared with commercially available epoxidized vegetable oil (Wypych, 2023). The acid value is an indication of the level of free fatty acid formed from the hydrolytic decomposition of glycerides to free fatty acid, which can be accelerated by an increase in oil extraction temperature and reaction temperature. The acid value of RSO and its epoxidized derivatives reduces from 42.49 to 5.09 ERSO-Ac and 27.91 ERSO-Fo (mg of KOH/g).

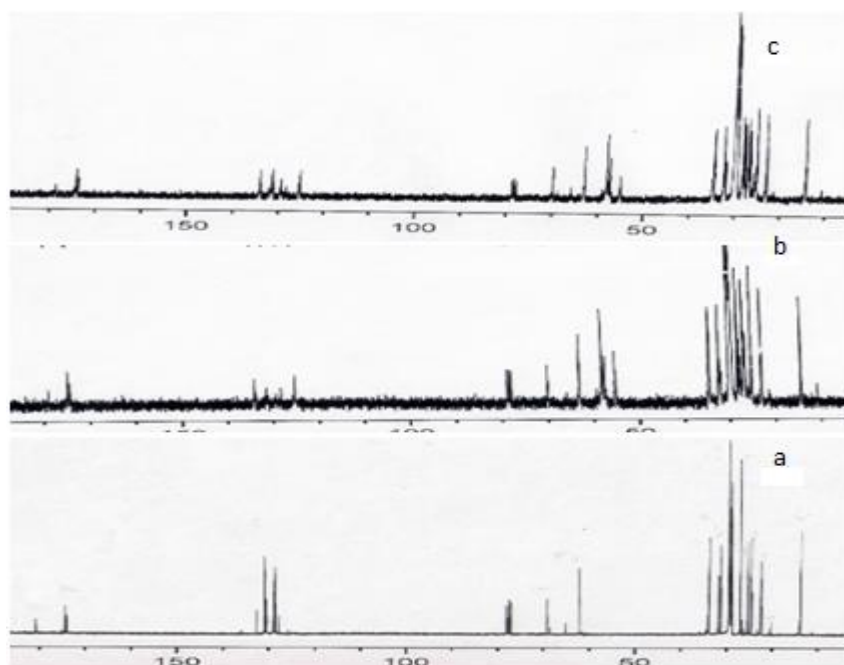


Fig. 3: ^{13}C NMR spectra of (a) RSO, (b) ERSO-Ac, and (c) ERSO-Fo

Table 2. Physico-chemical properties of modified rubber seed oil.

Properties	ERSO-Ac	ERSO-Fo	*ELO7190 Vikoflex®	*ESO 7170 Vikoflex®
Appearance	yellow	yellow	-	-
Specific gravity (30°C)	0.964	0.928	1.03	0.993
Acid value (mg KOH/g)	5.09	27.91	0.5 Max	0.5 Max
Saponification value (mg KOH/g)	210.8	201.94	-	-
Iodine value (g I ₂ /100g)	12.5	18.11	3.5 Max	2 Max
% Oxirane determined	4.70	7.04	9.0 Min	6.8 Min

*(Wypych, 2023)

Similarly, the saponification values of epoxidized RSO are approximately the same with neat RSO. This implied that the introduction of the oxirane group does not necessarily improve the saponification of the epoxides. The iodine value is a measure of unsaturation of triglyceride oil. The iodine value of 134.5 g I₂/100 g of oil obtained for RSO in this study is far higher than the values of 12.5 and 18.11 g I₂/100 g obtained for ERSO-Ac and ERSO-Fo, respectively, using the Wijs method. The difference in the iodine values of RSO and its derived products indicates that the double bond on the carbon atoms of the unsaturated fatty acid chains of rubber seed oil has been converted to an oxirane ring. The presence of a very low iodine value in the two products indicates there are residual unbroken double bonds. The relative conversion of unsaturated triglyceride oil to epoxidized oil based on available iodine values determined shows that epoxidation of RSO with acetic acid gave approximately 90.7 % conversion compared with formic acid, which resulted in 86.5 % yield of epoxidized oil.

Epoxidation of RSO with acetic acid and formic acid generally reduced the degree of unsaturation in the oil, which resulted in lower iodine values observed for RSO-Ac and ERSO-Fo. Reduction in iodine value of the ERSO-Ac and ERSO-Fo as a result of the chemical modification is one of the indications of successful epoxidation of the RSO (Okieimen *et al.*, 2005). The highest epoxy content observed in epoxidized RSO was 7.04 wt %; this value is comparable to the epoxy content observed in commercially available epoxidized soybean oil (Wypych, 2023) or epoxidized linseed oil (Table 2). The theoretical maximum oxirane oxygen content obtained for rubber seed oil is 8.48%. This value of theoretical maximum oxirane oxygen for epoxidized rubber seed oil is higher than the experimental values of 7.0 and 4.7% obtained for formic acid and acetic acid, respectively. These experimental values translate to a relative conversion to oxirane content of 83% for formic acid and 55% for acetic acid. The value of % oxirane of ERSO-Ac of 4.7 is lower than that 7.0 reported for ERSO-Fo. A similar observation

was reported in an earlier work (Petrović *et al.*, 2002). It is expected that the lower iodine value of 12.5 g I₂/100g and the corresponding iodine conversion of 90% observed in ERSO-Ac would be accompanied by a higher oxirane value compared with epoxidation with formic acid. However, the reverse case observed may have been due to the presence of inorganic acid used as the catalyst in epoxidation with acetic acid. Earlier reports showed that the mineral acid catalyzed the competing cleavage reactions resulting in hydroxyl and/or acetoxy compounds followed by dimerization (Petrović *et al.*, 2002). Also, the exothermic nature of the epoxidation reaction has been shown to lead to substantial rise in temperature and thus triggered undesirable side reactions (Gan *et al.*, 1992). Similarly, the lower iodine values obtained for the epoxidized rubber seed oil and the level of epoxide content further corroborate the disappearance of unsaturation at the stretching frequency of non-conjugated unsaturation at 3009 cm⁻¹ and the stretching frequency of alkene at 1654 cm⁻¹ in the FTIR spectra of ERSO-Ac and ERSO-Fo.

Conclusion: Epoxidized rubber seed oil with high epoxide content was produced by *in situ* epoxidation of rubber seed oil (RSO) with peracetic acid or performic acid. The epoxidized oil samples were carefully characterized and compared. The synthesis of epoxidized oils was deemed successful based on the reduction in iodine number of the oil compared with the epoxides. Study of IR and NMR spectra provides a strong proof of a better and optimum epoxide content. Thus, rubber seed oil and its derivatives have great potential as raw materials for production of bio-based polymers, which are currently underutilized. This versatile and environmentally friendly precursor for biopolymers could revolutionize manufacturing industries and sustainable future developments.

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Declaration of Conflict of Interest: The authors declare no conflict of interest.

Data Availability Statement: Data are available upon request from the author.

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