

## COMPARISON OF USUAL AND IN SITU ROUTES FOR SOYBEAN OIL EPOXIDATION

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**Abstract:** The epoxide is an important intermediate for preparation of chemically modified lubricants from the vegetable oils. Finding simple epoxidation ways, will reduce time and cost of lubricant preparation. In this paper the epoxidation reaction of soybean oil has been followed in two different conditions. In first circumstances, the reaction has been followed by initial preparation of peracid followed by its addition to the oil. In second route, the peracid has been produced in situ. By measurement of concentrations of double bonds and oxirane rings in one gram of the oil, as well as the acid value, the first conditions has been monitored and optimized corresponding to time, temperature and stirring rate. By same measurements, the second setting has been optimized relative to oil: acid: peroxide ratio, temperature, time, and stirring rate. Based on the obtained results, the advantages of in situ method relative to normal route have been proved. FTIR and NMR spectra of obtained epoxy under optimum in situ conditions has been recorded and compared with the corresponding spectra of initial oil.

**Key words:** Epoxidation, soybean oil, optimization, in situ, FTIR, NMR, lubricants.

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**Introduction:** Soybean oil is a triglyceride that typically contains 14% stearic, 23% oleic, 55% linoleic, and 8% linolenic acid [i, ii]. It is a potential low cost, renewable alternative for petroleum derived chemicals for a number of lubricant applications. However, it is well recognized that soybean oil has a variety of physical properties that limits its use in lubricant formulation [iii]. The limitations also exists for other vegetable oils. Chemical modification of the seed oils at the unsaturated bond sites results in changes to the physical properties of the substrate and allows products to be tailored to meet specific applications[iv]. Because the epoxide or oxirane group can easily be functionalized, epoxidized soybean oil as well as other epoxidized vegetable oils are promising intermediates for chemical adjustment of the oils [v]. This three member ring provides a more energetically favorable site for reaction. So, the production of derivatives that would be difficult to obtain directly from the unsaturated bond, will be attained from the epoxide intermediate [iv]. Also, epoxides are used directly as plasticizers, hydrolytic stabilizer, and plastic stabilizers [vi-viii]. Even though many

investigations related to epoxidized vegetable oils has been achieved [ix-xii]. Due to well-known significance of these compounds, it seems that further study on the different routes of their synthesis is essential. Finding the easiest and most economic synthesis methods will be the aim of such researches. The low price production and extension of the usage of bio based lubricants will be the result of such studies. In this paper two ways for epoxidization of soybean oil are explained and compared with each other. This work is in continue of our other investigations in the lubricants field [xiii-xviii].

### **Experimental:**

**Materials:** All of the materials were used without any further purification. The used materials are: glacial acetic acid from Merck Company, Soybean oil from Ina Company, hydrogen peroxide from Aldrich Company, 32% HBr in glacial acetic acid from Merck Company, liquid bromine from Acros Company, carbontetrachloride from Merck Company, and crystal violet from Aldrich Company.

**Instrumental:** FTIR spectra were recorded on a JASCO 6300 FT-IR system in a scanning range of 400-4000  $\text{cm}^{-1}$  for 32 scans at a spectral resolution of 4  $\text{cm}^{-1}$  with a pair of KBr crystals in thin film.  $^1\text{H}$  NMR spectra were recorded using a Bruker Avance III 400 NMR, using a 5 mm broadband inverse Z-gradient probe in  $\text{CDCl}_3$ , and Bruker Icon NMR software was used. Peaks were referenced to 3-tetramethylsilane- $\text{d}_4$  (TMS) at 0.0000 ppm. Each spectrum was Fourier transformed, phase corrected, and integrated using ACD spectrum manager.

**Determination of saponification value:** A mixture containing 1 gr of oil sample and 25 mL of 0.5 N of ethanoic potassium hydroxide was refluxed for one hour. Then it was titrated at 70°C with 0.5 normal of hydrochloric acid using phenolphthalein indicator. A blank was run in the same manner. Finally, the saponification value was calculated from the multiplication of the difference between consumed volume of sample and blank, by 28.05 [19]. The obtained value was used for estimation of molecular weight of the oil.

**Determination of iodine value:** It is a measure of the unsaturations of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample. Briefly, the sample was dissolved in appropriate amounts of carbontetrachloride and the solution was titrated with standard solution of bromine in carbontetrachloride (1.52 molar). The end point of the reaction was determined by observing the permanent color of bromine in the oil containing solution. By multiplication of the consumed volume of bromine solution by 1.52 and then multiplication of the resulting value by 1.41 (the ratio of molecular weight of iodine to bromine), the iodine value is determined.

**Determination of double bonds:** the no. of milimoles of double bonds was determined by the same route of iodine value measurement. However, the desired value was obtained by multiplication of consumed volume of bromine solution by 1.52.

**Determination of acid value:** It is defined as the mass of potassium hydroxide in milligrams that is required to neutralize one gram of the sample. This was determined following AOCS standard method, AOCS Ca 3a – 63 [xix]. Briefly, certain amount of the sample was dissolved in hot ethyl alcohol and titrated with KOH solution using phenolphthalein as indicator. The required amount of KOH (mg) per gram of the sample, is the acid value of the sample.

**Determination of epoxide:** This was determined following AOCS standard method, AOCS Cd 9 – 57. Briefly, one gram of the sample was dissolved in appropriate amounts of glacial acetic acid and titrated with HBr solution in glacial acetic acid using crystal violet as indicator. The no.

of mmoles of epoxide is calculated from the multiplication of 0.53 (molarity of HBr solution) by the consumed volumes of HBr.

**Epoxidation under normal conditions:** This was followed in two steps. In the first step 286 mL of glacial acetic acid, 1.7 mL sulfuric acid, and 45 mL hydrogen peroxide were transferred to a dry three neck 500 mL beaker. The mixture was stand at room temperature for 20 hours. In the next step 40 mL of the recent solution was added to 10 gr of soybean oil and the epoxidation was followed in reflux and non reflux conditions. The recent reaction was optimized according to time, temperature and stirring rate. The obtained product of each experimental set was separated by washing with 5% solution of sodium bicarbonate, extraction by organic solvent and drying under vacuum. The final product of each step was employed for determination of epoxy content, double bond concentration and acid value. The optimization results are given in the section of results and discussion.

**Epoxidation under in situ conditions:** In a dry three neck 500 mL round-bottom flask fitted with a condenser and equipped with a thermometer, 44 gr of soybean oil and 6 gr of glacial acetic acid were mixed. To the recent mixture, appropriate amounts of H<sub>2</sub>O<sub>2</sub> 30% was added drop by drop. In the next step, 0.2 gr of H<sub>2</sub>SO<sub>4</sub> catalyst was added to the blend. While stirring, the mixture was heated. In continue the muddle was cooled to room temperature and washed with hot solution of sodium bicarbonate and then water, for three times. The resulting aqueous and organic phases were separated and used for next steps. Then, the organic phase was dried with MgSO<sub>4</sub>. In order to obtain optimum conditions of stirring rate, reaction time, temperature, and ratio of oil: acid: peroxide, the whole process was replicated at several new setting. In each experiment, the double bond and epoxy concentration, as well as acid value of the adduct was measured. The optimum conditions were recognized by the comparison of test results of different trials. The final obtained product under optimized conditions was characterized by FTIR and <sup>1</sup>H NMR techniques

**Results and discussion:** General properties of the soybean oil as well as the corresponding test methods are given in Table 1. By calculation of ester value via the subtraction of acid value from saponification value, and using the equation: Molecular weight = (3000 × 56.1/ester value), the average molecular weight of the triglyceride has been obtained as 851.3. Then, by using the iodine value, the no. of mmoles of double bonds in one gram of the used oil has been calculated as 4.89. In continue by division of the recent quantity to the no. of mmoles of the triglyceride in one gram of the oil, the average no. of double bonds in one molecule of triglyceride has been calculated as 4.16. These double bonds are mainly due to existence of 18:1, 18:2, and 18:3 fatty acids in the structure of the triester. Thus, the complete epoxidation involves the formation of 4.16 oxirane ring in each molecule.

Normal method was followed by initial preparation of peracid (Eqn. 1) followed by

$$\text{CH}_3\text{CO}_2\text{H} + \text{H}_2\text{O}_2 \rightleftharpoons \text{CH}_3\text{CO}_3\text{H} + \text{H}_2\text{O} \quad (1)$$

addition of peracid to the oil. In order to obtain the best reaction conditions the reaction was optimized relative to temperature, time and stirring rate. The optimization was carried out by tracing the reaction at different experimental conditions. The tracing was performed by measurement of no. of the mmoles of epoxy groups, double bonds and acid value in one gram of the oil.

It is expected that during the reaction progress, the no. of mmoles of epoxy to be increased and the no. of mmoles of double bonds to be decreased. On the other hand, observation of a fixed acid value indicate that during the the reaction progress, conversion of triglyceride to

free acid has not been occurred. Equality of the diminished amount of the no. of mmoles of double bonds with increase of the no. of the mmoles of epoxy groups, beside a fixed acid value, designates that all of the double bonds have merely been converted to epoxy groups. Inequality indicate that the side reaction has been occurred.

The optimization was started from the temperature as most important agent and completed with time and stirring rate as the second and third significant factors. The sequence of importance was identified by random changes of each factor and visual observation of viscosity and color change as approximate index of epoxy formation. Based on the several experiments a rule of thumb was found that: epoxy formation is along with increase in viscosity and fading of yellow color.

The results of temperature optimization in normal conditions are given in Table 2. All of the exposed data are due to one gram of the oil. Total double bonds (TD) is the no. of consumed mmoles of standard solution of bromine in carbontetrachloride (1.52 molar), for complete saturation of the soybean oil; unreacted double bonds (URD) is the amount of consumption for reacted oil; the difference between the two recent parameters is reacted double bond (RD); the epoxidized double bond (ED) is the consumed mmoles of standard solution of HBr for ring opening of epoxide; the difference between RD and ED is unepoxidized double bond (UED) and indicate the amount of saturation, via reactions other than epoxidation; the percentages of epoxidation (ED%) , side reactions (SR%) and unreacted double bonds (URD%) has been obtained by division of ED, UED and URD to TD value. As the data indicate (Table 2), upon raising the temperature, the RD value increases systematically. On the other hand it is observed that ED and RD values are not equal, which means that beside oxidation, side reaction has also been occurred. According to the reaction conditions it seems that acetylation and diol formation are the possible side reactions.

An overview to the data of Table 2 indicates that considerable epoxidation has not been occurred, and at best temperature conditions (i. e. 60°) only 22.5% epoxidation has been occurred. The fixation of acid value (AV), designates that during the reaction considerable cleavage of triglyceride to free acid has not been occurred. However, a sudden increase of AV is observed at 100 c. Which means that at recent temperature a high degree of triglyceride cleavage has been occurred. Moreover at 100 c the lowest ED% is obtained. Thus, following the reaction at recent conditions is not along with ideal results.

For obtaining the best time under normal circumstances, at the most favorable temperature of 60°c, the reaction was performed at 2, 5, and 10 hours. The stirring rate was 700 rpm (Table 3). As it is seen, the best results have been obtained after 2 hours. At longer times ED% is decreased and the acid value raises up. Probably at longer times the oxirane ring is opened by existing acid or water in the reaction media.

The most excellent stirring rate for normal setting was obtained by monitoring the reaction at the optimum temperature of 60°c and the finest time of 2 hours. The obtained results are given in Table 4. As it can be seen, at the stirring rate of 700 rpm the highest ED% and the lowest URD is obtained. Which means that the recent stirring rate is the most suitable one.

The final results of optimization at normal conditions indicate that the maximum reachable ED% is 22.5, which is not a considerable value. In addition, under these circumstances the SR% value is high. Consequently, this method cannot be regarded as an ideal one.

In order to obtain the best in situ circumstances, the experimental conditions were optimized according to the oil to oxidant to peroxide ratio, temperature, time and stirring rate. The sequence of optimization was ratio, temperature, time and stirring rate.

The obtained results for in situ conditions and at oil: acid: peroxide ratios of 1: 2: 10, and 1: 2: 20 and at constant temperature of 60 °c, steady time of 30 hours and stirring rate of 700 rpm, are given in Table 5. Looking at the data indicate that 1:2: 20 is the best ratio. So, this ratio was obtained for the rest of the work.

The results due to determination of best temperature for in situ conditions are set in Table 6. For temperature optimization, the selected ratio of oil: acid: peroxide as well as constant time of 30 hours, and stirring rate of 700 rpm were employed. According to the data, the optimum temperature of 60 °c is confirmed.

The obtained data due to determination of the best time under in situ conditions are given in Table 7. The numbers have been gained in the optimum ratio and temperature conditions and at constant stirring rate of 700 rpm. The figures proves that 6 hours is the most suitable time.

Finally at optimum determined ratio, temperature, and time, the best stirring rate was determined (Table 8) as 700 rpm.

Comparison of the results of in situ and normal conditions specifies that under normal conditions, the maximum attainable ED% is 22.5%. However, in the case of in situ ED% up to 93.7 can be obtained. The SR% value under in situ conditions is not more than 0.8%. While in normal conditions it is about 40%. The lower AV and URD% are the other advantages of in situ conditions. The higher reaction time is the main disadvantage of in situ method.

FTIR spectrum of soybean oil is given in Fig. 1. As it can be seen, the methylene band is appeared at 723 cm<sup>-1</sup>. The peak at 1376 cm<sup>-1</sup> can be attributed to symmetric methylene and the 1463 cm<sup>-1</sup> peak can be assigned to unsymmetrical methylene. Unsymmetrical stretching of ester is appeared at 1163 and 1239 cm<sup>-1</sup>. The double bond peaks are seen in 1654 cm<sup>-1</sup>. The observation of a peak at 1745 cm<sup>-1</sup> can be attributed to carbonyl group. The appearance of a peak at 3009 cm<sup>-1</sup> can be assigned to stretching of unsaturated CH [xx, xxi].

The comparison of IR spectrum of epoxide, (Fig. 1, bottom) with that of soybean oil (Fig. 1, top), indicate that the stretching CH double bond which in the soybean oil spectrum can be seen at 3009 cm<sup>-1</sup>, has been disappeared in epoxide spectrum. In addition, the peak at 1654 cm<sup>-1</sup> which is due to double bond has been disappeared in the epoxide spectrum. Based on the IR information, the creation of epoxide is fairly confirmed.

<sup>1</sup>H NMR spectrum of soybean oil is shown in top section of Fig. 2. The chemical shift of 0.885 to 0.830 ppm can be assigned to terminal CH<sub>3</sub>. The methylene protons are appeared at 1.228 to 1.323 ppm. β- CH<sub>2</sub> of carbonyl group can be seen at 1.5 ppm. The appearance of chemical shift at 1.989-2.030 ppm can be attributed to allylic protons. The α-protons of carbonyl group are appeared at 2.313-2.257 ppm. The chemical shift at 2.723-2.761 ppm is due to bisallylic protons. The four protons of methylene backbone are appeared at 4.06-4.145 and 4.242-4.296 ppm. A methyne proton which exists in the backbone of triglyceride molecule has been appeared at 2.276-5.525 ppm. Olephinic protons have been appeared at 5.288- 5.368 ppm in a multiple form [20, 21].

NMR spectrum of epoxide is given in bottom section of Fig. 2. The spectrum shows that the epoxy protons have been appeared in 3.0-3.2 ppm, methyne protons of CH<sub>2</sub>-CH-CH<sub>2</sub> of sample skeleton are emerged at 5.1 to 5.3 ppm. Methylene protons of CH<sub>2</sub>-CH-CH<sub>2</sub> of sample skeleton are appeared at 4 to 4.4 ppm. The CH<sub>2</sub> protons which are bonded to epoxide are seen at 2.8-3.0 ppm. The CH protons which are bonded to epoxide are shown up at 3.0 -3.2 ppm. α-CH<sub>2</sub> of carbonyl group has become visible in 2.2-2.4 ppm. α-CH<sub>2</sub> of epoxy group has been appeared in 1.7-1.9 ppm. β-CH<sub>2</sub> of carbonyl group come into viewed at 1.55-1.70 ppm. β-CH<sub>2</sub> of epoxy group has been appeared at 1.4-1.55 ppm. The saturated methylene has been come out at 1.1-1.4

ppm, and the terminal CH<sub>3</sub> have been appeared in 0.8-1 ppm. The evidences of NMR spectrum is a further confirmation of epoxide formation.

### Conclusions:

- 1) The optimum conditions for epoxidation of soybean oil at normal conditions are 60 °C, 2 hours and stirring rate of 900 rpm.
  - 2) The maximum percentage of epoxidation under normal conditions is 22.5%.
  - 3) Under normal conditions, epoxidation is along with considerable side reactions.
  - 4) The optimum conditions for epoxidation under in situ conditions are the ratio of oil: acid: peroxide of 1:2:20, temperature of 60 °C, reaction time of 30 hours and stirring rate of 900rpm.
  - 5) The maximum attainable ED% under in situ conditions is 93.7.
  - 6) Because of higher yield, lower side reaction, and lower acid value, the in situ is preferred to normal method. However this method does have the disadvantage of higher reaction time.
- The recent defect should be solved by finding the better catalysts.

**Acknowledgement:** The financial support of this work by research council of Shahrekord University is highly appreciated. The authors were also partially supported by the Center of Excellence for Mathematics, University of Shahrekord.

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Received on August 8, 2012.

<b>Property</b>	<b>Test method</b>	<b>Value</b>
Soaponification value (mg KOH/ gr oil)	ASTM D-5558-5	199.3
Acid value (mg KOH/ gr oil)	AOCS Ca 3a – 63	1.6
Ester value (mg KOH/ gr oil)	-	197.7
Average molecular weight	-	851.3
Iodine value	AOCS Cd 1– 25	123.7
Milimoles of double bonds per one gram of the oil	AOCS Cd 1– 25	4.89
Double bond per molecule	-	4.16

**Table 1** General properties of soybean oil.



T (°C)	<sup>a</sup> TD	<sup>b</sup> RD	<sup>c</sup> URD	<sup>d</sup> ED	<sup>e</sup> UED	<sup>f</sup> ED%	<sup>g</sup> SR%	<sup>h</sup> URD%	<sup>i</sup> AV
25	4.89	1.89	3.00	0.44	1.45	9.0	29.7	61.3	3.4
45	4.89	2.63	2.25	0.88	1.75	18.0	35.8	46.2	3.4
60	4.89	3.00	1.89	1.10	1.90	22.5	38.9	38.6	3.4
100	4.89	1.51	3.38	0.22	1.29	4.5	26.3	69.1	134.6

**Table 2** Monitoring of epoxidation of soybean oil at different temperatures and non in situ conditions. In all cases the reaction time and stirring rate were, two hours and 700 rpm, respectively.

a) total double bonds; b) reacted double bonds; c) unreacted double bonds; d) epoxidized double bonds; e) unepoxidized double bonds; f) percentage of epoxidation; g) percentage of side reactions; h) percentage of unreacted double bonds; i) acid value.

<b>Time (hour)</b>	<b>TD</b>	<b>RD</b>	<b>URD</b>	<b>ED</b>	<b>UED</b>	<b>ED%</b>	<b>SR%</b>	<b>URD%</b>	<b>AV</b>
2	4.89	3.00	1.89	1.10	1.90	22.5	38.9	38.6	3.4
5	4.89	3.39	1.50	0.5	2.89	10.2	59.1	30.7	17.0
10	4.89	3.76	1.13	0.33	3.43	6.75	70.1	23.1	30.6

**Table 3** Monitoring of epoxidation of soybedan oil at different times and non in situe conditions. In all cases the temperature and stirring rate were 60°c and 700 rpm, respectively.

<sup>a</sup> SR	TD	RD	URD	ED	UED	ED%	SR%	URD%	AV
350	4.89	2.45	2.44	0.79	1.66	16.1	33.9	49.9	3.4
700	4.89	3.00	1.89	1.10	1.90	22.5	38.9	38.6	3.4

**Table 4** Monitoring of epoxidation of soybean oil at 700 and 900 rpm as well as non in situ conditions. In both cases the temperature and time were 60 °c and 2 hours, respectively.  
a) stirring rate.

<b>Ratio</b>	<b>TD</b>	<b>RD</b>	<b>URD</b>	<b>ED</b>	<b>UED</b>	<b>ED%</b>	<b>SR%</b>	<b>URD%</b>	<b>AV</b>
1:2:10	4.89	2.25	2.64	2.18	0.07	44.6	1.4	54.0	1.7
1:2:20	4.89	4.59	0.3	4.58	0.01	93.7	0.2	6.1	1.7

**Table 5** In situ epoxidation of soybean oil at two different oil:acid:peroxide ratios. In both cases the temperature, time, and stirring rate, were 60°C, 30 hours and 700 rpm, respectively.

<b>T (°c)</b>	<b>TD</b>	<b>RD</b>	<b>URD</b>	<b>ED</b>	<b>UED</b>	<b>ED%</b>	<b>SR%</b>	<b>URD%</b>	<b>AV</b>
25	4.89	1.54	3.35	1.50	0.03	22.7	0.6	68.5	1.7
45	4.89	2.31	2.58	2.27	0.04	46.8	0.8	5.24	1.7
60	4.89	4.59	0.3	4.58	0.01	93.7	0.2	6.1	1.7

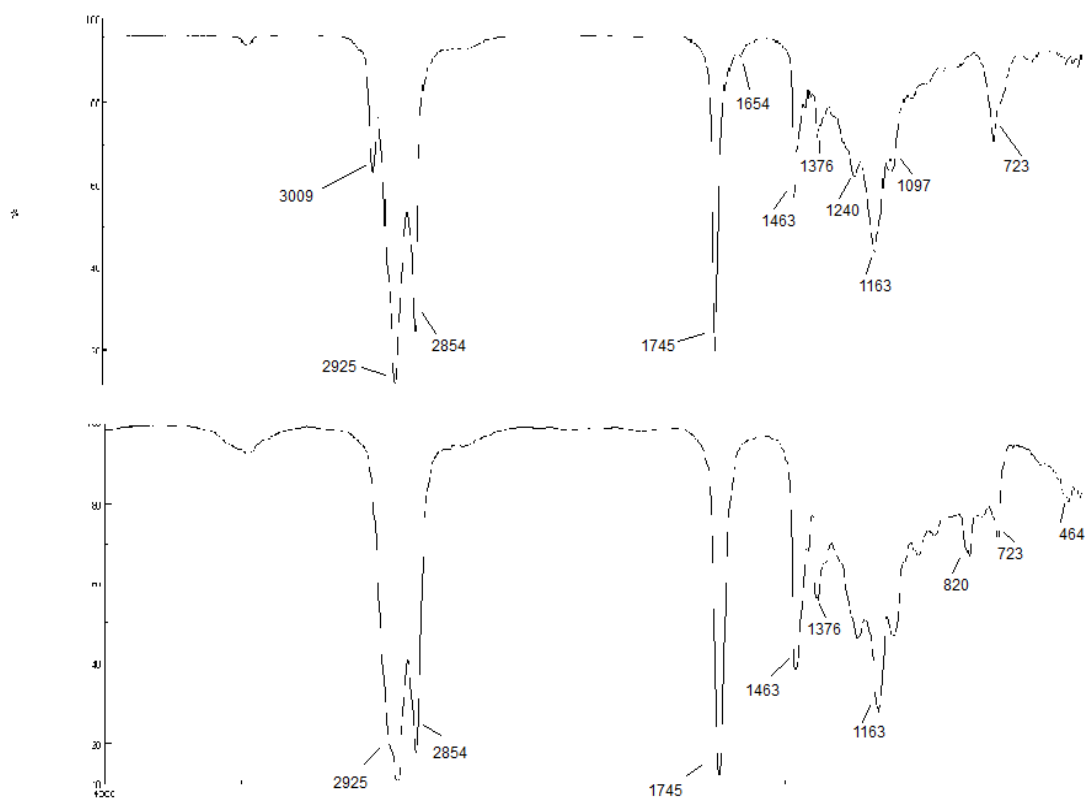
**Table 6** In situ epoxidation of soybean oil at different temperatures. In all cases the oil:acid:peroxide ratio is 1:2:20, the time is 30 hours and the stirring rate is 700 rpm.

<b>T (°c)</b>	<b>TD</b>	<b>RD</b>	<b>URD</b>	<b>ED</b>	<b>UED</b>	<b>ED%</b>	<b>SR%</b>	<b>URD%</b>	<b>AV</b>
5	4.89	2.99	1.90	2.97	0.02	60.7	0.4	38.9	1.7
10	4.89	3.87	1.02	3.85	0.02	78.7	0.4	20.8	1.7
20	4.89	4.29	0.60	4.25	0.04	86.9	0.8	12.2	1.7
30	4.89	4.59	0.30	4.58	0.01	93.7	0.2	6.1	1.7

**Table 7** In situ epoxidation of soybean oil at different times. In all cases the oil:acid:peroxide ratio is 1:2:20, the temperature is 60° c, and the stirring rate is 700 rpm.

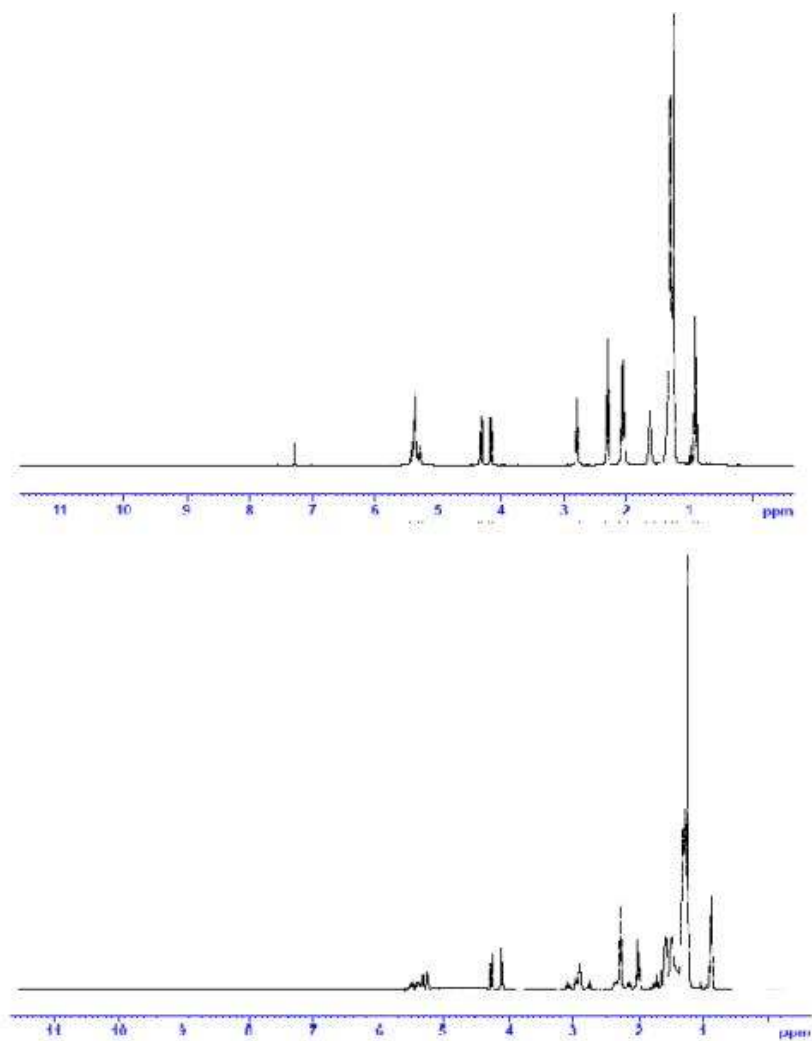
<b>SR</b>	<b>TD</b>	<b>RD</b>	<b>URD</b>	<b>ED</b>	<b>UED</b>	<b>ED%</b>	<b>SR%</b>	<b>URD%</b>	<b>AV</b>
700	4.89	3.49	1.40	3.44	0.05	16.1	1.0	28.6	1.7
900	4.89	4.59	0.3	4.58	0.01	93.7	0.2	6.1	1.7

**Table 8** Monitoring of epoxidation of soybean oil at 700 and 900 rpm as well as non in situ conditions. In both cases the temperature and time were 60°c and 2 hours, respectively.



**Fig. 1** FTIR spectra of soybean oil (top) and epoxidized soybean oil (bottom)





**Fig. 2** NMR spectra of soybean oil (top) and epoxidized soybean oil