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# EPOXIDIZED SUNFLOWER OILS AND THEIR RESEARCH

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**Abstract.** In this study, the epoxidation of sunflower oil is studied. Sunflower oil was oxidized with hydrogen peroxide in the presence of Ant acid. The process was carried out in a flask equipped with a mixer and a thermometer. Sunflower oil was oxidized with 30% hydrogen peroxide in the presence of Ant acid. Tetrabutyl titanate was used as a catalyst. To remove excess Ant acid and hydrogen peroxide after the reaction was completed, the reacting mixture was washed using a sodium carbonate solution and separated from the aqueous solution using a separation funnel. It was then washed several times with distilled water and dried in a vacuum. The composition and structure of the resulting epoxidized sunflower oil was studied and analyzed using the methods of modern IQ spectroscopy, raman spectroscopy, NMR and PMR. It was also found to have an iodine number and an epoxy number. In IQ-spectroscopic analysis, the formation of the epoxy group was demonstrated by the presence of fluctuations in the area 2966.52-3055.24 $cm^{-1}$. Sunflower oil has an iodine number of 124.6, and when it was oxidized with hydrogen peroxide with an oxide, it was found that the number of iodine in it changed to 117.8.

**Keywords:** sunflower oil, epoxidation, formic acid, hydrogen peroxide, IR- spectrum, Raman spectrum, NMR and PMR.

## 1. Introduction

Vegetables oils are a sustainable and renewable raw material resource extracted from plants and wood. These vegetables oils are used like starting material and they offers numerous advantages such as low toxicity and inherent biodegradability. In the last years, the epoxidation of vegetables oil received a great interest from industry [1]. Epoxidation is a reaction widely used to form oxirane rings from ethylenic unsaturations (C=C) [2]. The cyclical structure of oxirane rings has a bond angle of 60°C, making them highly strained and highly reactive [3]. Classic methods employed for the oxidation of vegetable oils use homogeneous catalytic processes that generate a lot of waste, corrode equipment and require large amounts of reagents. Heterogeneous catalysts have the advantage of easy separation and recycling of the catalyst. For this reason, ion exchange resin has been studied to promote epoxidation of vegetal oils [4]. MCFs can be made from vegetable oils, but some oils have low stability, mainly at high temperature. To solve this problem, epoxidation reactions are applied to convert unsaturations to oxirane rings followed by opening of the rings with water, to form the vicinal diol [5,6]. Several authors have investigated heterogeneous catalysts based on the sulfonation of incomplete pyrolyzed biomass, such as sucrose [7], glucose [8,9] and biochar [10]. Conventionally, the production of epoxidized vegetable oils is carried out by the Prileschajew method [11]. It is a liquid–liquid reaction system, where there are several consecutive and parallel exothermic reactions [12].

Sepulveda et al. [13] tested different alumina catalysts for the epoxidation of methyl oleate and soybean oil methyl ester by hydrogen peroxide in different organic solvents. They reached a conversion of ca. 100%, Di Serio et al. [14] tested $Nb_2O_5$–$SiO_2$ catalyst for the epoxidation of soybean oil in organic solvents, but the selectivity was quite low, Turco et al. [15] studied the epoxidation of soybean oil and methyl oleate with hydrogen peroxide on $\gamma$-alumina in the presence of different organic solvents. They have demonstrated that the solvent plays an important role for this system. They have reported that acetonitrile was the best solvent for the epoxidation of methyl oleate, Parada Hernandez et al. [16] have studied the system $H_2O_2$/alumina/ethyl acetate for the epoxidation of methyl ricinoleate. They demonstrated that this system is efficient for the epoxidation of this oil.

In the present study, epoxidation and ozonolysis led to a decrease of 40.8 and 51.9 % in the degree of unsaturation of corn oil, respectively [17]. Epoxidation of flax oil in a conjugate system with hydrogen peroxide using chlorinated cation exchanger KU-2×8 as catalyst was studied. The influence exerted on the epoxidation rate and target product quality by the temperature, stirring intensity, catalyst amount, and reactant ratio was examined [18]. This study presents epoxidizing *podocarpus falcatus* seed oil through epoxidation reaction by using synthesized solid sulfonated silica catalyst from locally available sugarcane bagasse ash [19]. Some of them are play an important role as corrosion inhibitors for technological tools [20-24].

Renewable resourced polymer composites from vegetable oils and bio-fibers are receiving increasing attention from various industries due to their characteristics of being less heavy, environment friendly, and biodegradable. Lignocellulosic natural fibers have immense potential to be used as reinforcing fillers due to their characteristics of being less expensive, abundant obtainability, lower density, higher specific strength and modulus, and good interfacial strength with thermoset polymers. In this chapter, epoxidized nonedible linseed and castor oils are proposed as a diluent to petro-based epoxy in formulating toughened bio-based copolymers. Unidirectional sisal fibers were reinforced within a network of such bio-epoxy copolymers in order to achieve an optimal stiffness–toughness balance. Cardanol based phenalkamine, a bio-renewable crosslinker, is used to develop well toughened sustainable and green composite materials. The composites were subjected to various thermal, mechanical, dynamic mechanical, and morphological tests to investigate the impact of nonedible epoxidized oils and sisal fibers in addition to the petro-based epoxy matrix. The present study shows the method for design and development of novel sustainable green composites with higher bio-source content (>65%) meant for shock absorbing applications [25-29].

The molybdenum(II) tricarbonyl complexes $[Mo(CO)_3I_2L_n]$ ($n$ = 1, L = 2,2′-bipyridine, 4,4′-di-*tert*-butyl-2,2′-bipyridine; $n$ = 2, L = pyridine, 4-*tert*-butylpyridine) have been examined as catalyst precursors for the epoxidation of the biorenewable olefin methyl oleate with *tert*-butylhydroperoxide.

In situ oxidative decarbonylation of the precursors gives highly active and selective molybdenum(VI) catalysts, which were identified as the one-dimensional molybdenum oxide/bipyridine polymer $[MoO_3$(2,2′-bipyridine)], octanuclear $[Mo_8O_{24}$(4,4′-di-*tert*-butyl-2,2′-bipyridine$)_4]$, and the pyridinium β-octamolybdates $(LH)_4[Mo_8O_{26}]$ for L = pyridine or 4-*tert*-butylpyridine [29-32].

The octamolybdate salt $(H_3biim)_4[\beta\text{-}Mo_8O_{26}]$ (**1**) has been prepared in good yield by hydrolysis of the complex $[MoO_2Cl_2(H_2biim)]$ ($H_2biim$ = 2,2′-biimidazole).

Compound **1** showed a good performance as a (pre)catalyst for the epoxidation of olefins using either *tert*-butylhydroperoxide (TBHP) or hydrogen peroxide as oxidant [33].

**The purpose of the study** is to study the process of epoxidation of sunflower oil with additives and the composition and properties of epoxidized oil using modern research methods.

## 2. Materials and Methods

Sunflower oil according to GOST 1.2-2009 and formic acid according to GOST 1706-78 are used in the experiment. For this, 50 g of sunflower oil and 7 g of formic acid were added to the flask, the glass was slowly heated while stirring at a speed of 200 rpm, and after 30 minutes, 15 g of 30% hydrogen peroxide was added. The temperature was maintained at 70°C for 3 hours. 4 g of hydrogen peroxide was added. Then, after 3 hours, hydrogen peroxide was added to the reaction mixture and stirred again. The resulting mixture was cooled, washed with 5% sodium bicarbonate aqueous solution, and separated in a separatory funnel. The organic phase was washed with distilled water. The water mixed with the organic phase is separated by vacuum and the oil is dried. The iodine number and epoxide number of the obtained epoxidized oil are determined. The resulting mixture was cooled and $CO_2$ gas was passed while heating in an oil bath by adding 5 grams to a test tube, the process was carried out in the presence of a catalyst. The process of epoxidation of formic acid, as well as large-ton vegetable oils: sunflower oil, was studied. Oxidation was carried out using hydrogen peroxide, and tetra butyl titanate catalyst, which were obtained using the appropriate concentrated acids and an aqueous solution of hydrogen peroxide. Oxidation kinetics was assessed by an accumulation of peroxide compounds, iodine value, and number.

## 3. Results and Discussion

***3.1.* The IR spectrum of epoxidized sunflower oil.** *IR*-spectrum ("IR Tracer-100" SHIMADZU CORP., Japan, 2017) analysis of the substance obtained as a result of the reaction was carried out. As can be seen from Figure 1, the presence of a plane (=C-H) and a ring (C=C) at 1506 $cm^{-1}$, the presence of a strain vibration (-C-H) at 1230 cm-1, and a

deformation vibration at 1031 $cm^{-1}$ (-O-$CH_3$) 771 $cm^{-1}$ (CH-O-CH-) was found to be present. The presence of bonds belonging to the (-C=C-) group in the 1581.63 - 1606.7 $cm^{-1}$ area, the presence of a stretching vibration in the 1182.36 - 1296 $cm^{-1}$ area (C-C-C), 1361.74 cm-1 The presence of bonds in the areas (-$CH_2$-$CH_2$-) indicates the composition of the original substances, the presence of bonds in the area 1456 $cm^{-1}$ (-O-$CH_2$-), the presence of an epoxy group in the areas 2966.52-3055.24 $cm^{-1}$ proves that sunflower oil is epoxidized (1-picture)**.**

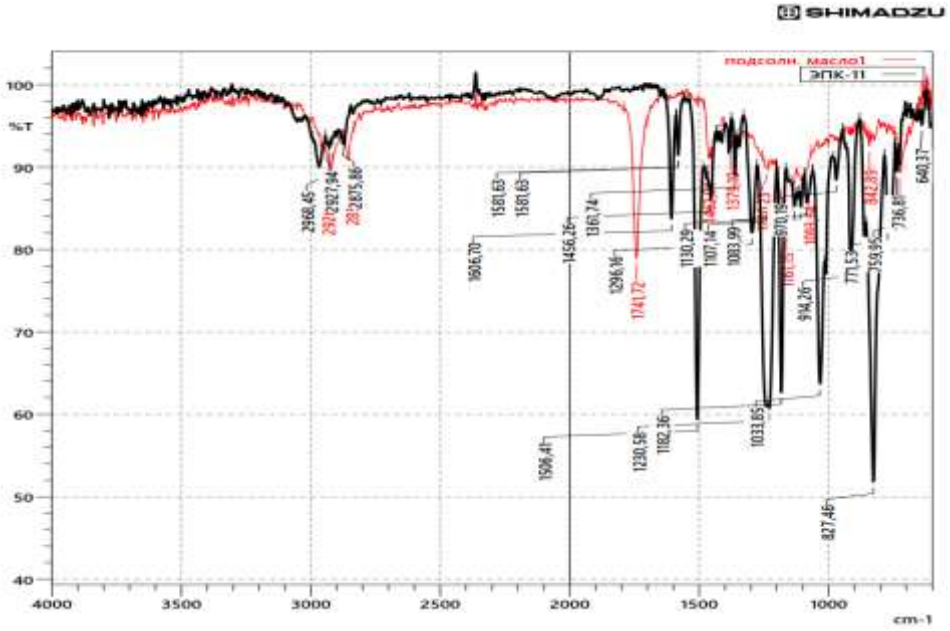



**Fig 1**. IR-spectrum of epoxidized oil based on sunflower oil.

**3.2. Roman Spectroscopy**. Analysis. In the following a pictur, Raman spectrum analysis of epoxidized sunflower oil was carried out. According to the results of the analysis, the vibrations of the C-H groups at the absorption frequency of 3078 $cm^{-1}$ at the frequency of 2929-1989-1694 $cm^{-1}$ are the branches of the $CH_2$ and $CH_3$ groups, and the presence of absorption bands in the region showed that this is not observed in non-epoxidized oil. 1183-1290 $cm^{-1}$ and 667-821 $cm^{-1}$ (CH-O-CH-) were studied to be present in the absorption zone in the region, characteristic of the vibrations of methylene groups, due to the increase in its intensity, the process is active oxygen released as a result of the decomposition of hydrogen peroxide The progress of oxidation reactions under the influence showed that it is epoxidized.

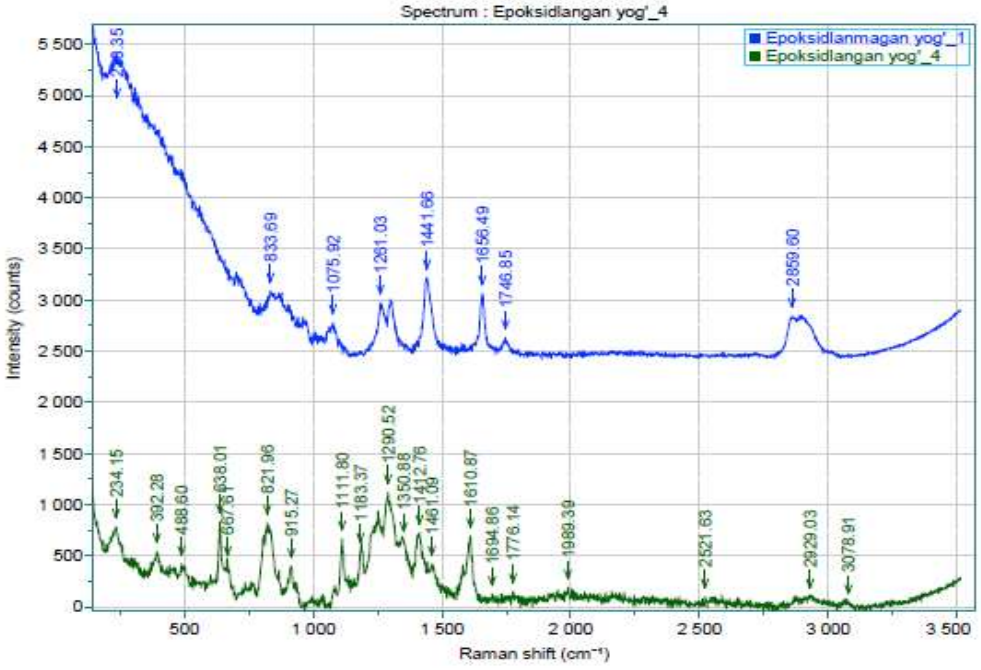
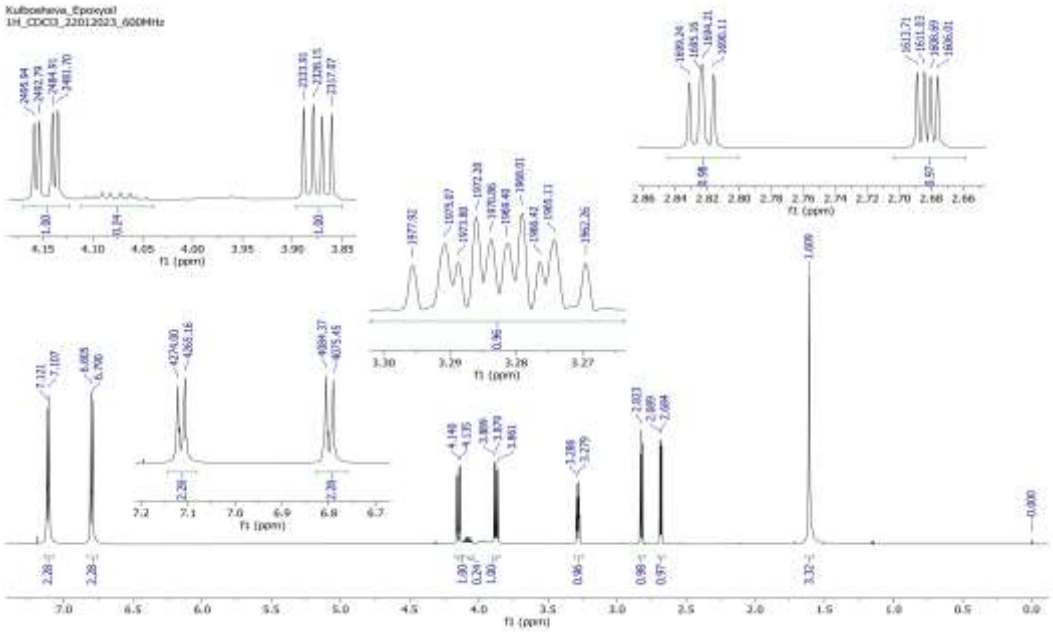
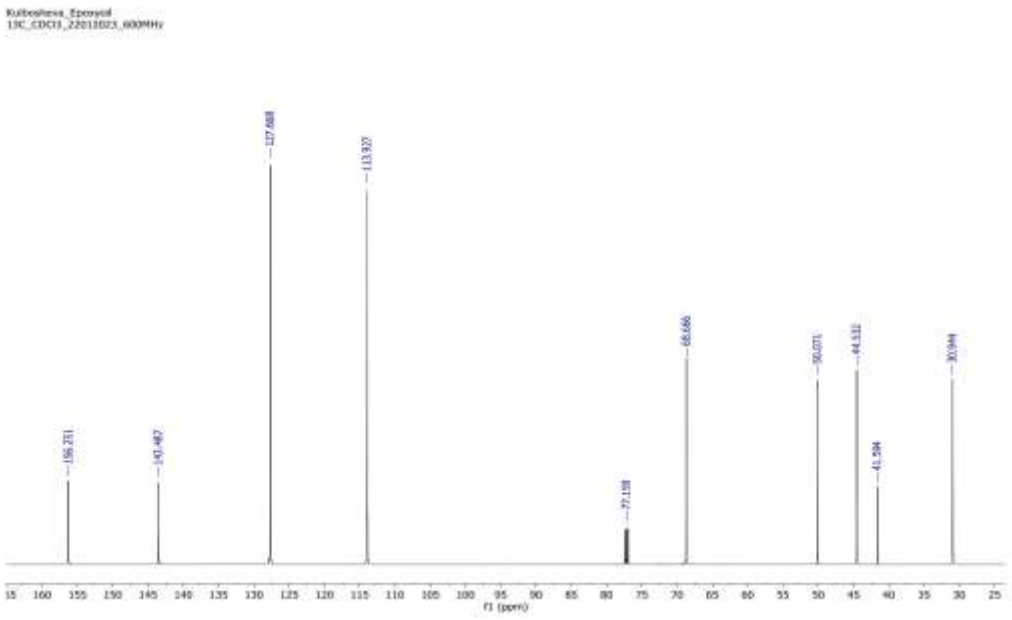



**Fig. 2.** Roman spectrum of epoxidized oil based on sunflower oil.

**3.3. NMR and PMR analysis of epoxidized sunflower oil.** NMR spectra were recorded on a JNM-ECZ600R spectrometer (JEOL, Japan) at an operating frequency of 600 MHz, for 1H in $CDCl_3$ solutions.



A



B

**Fig. 3.** NMR (A) and PMR (B) analysis of the obtained epoxidized sunflower oil.

TMS (0 ppm) was used as an internal standard in the $^{1}H$ NMR spectra. In the $^{13}C$ NMR spectra, the chemical shift of the solvent (CDCl3, 77.16 ppm relative to TMS) was used as an internal standard. 1, 1' in YaMR analysis; 2, 2'; 3, 3'; 4, 4'; 5¸ 5'; 6, 6'; 8, 8'; 9, 9'; 10, 10'; 11, 11'; Signals of 12 carbons are respectively 143.49; 127.67; 113.93; 156.25; 113.93; 127.67; 68.67; 50.07; 44.53; 30.94; 41.59 m.u. observed in the fields. In the PMR spectrum, doublet-doublet signals of protons located in symmetric 8, 8' and 10, 10' - symmetric carbon atoms in the epoxidized sunflower oil molecule are 3.87-4.15 m.u, respectively. and 2.68-2.82 m.u. in the field and 2, 2'; 3, 3'; 5¸ 5'; Signals of protons located in 6, 6'-carbon atoms are respectively 7.11; 6.80; 6.80; 7.11 m.u. observed in the fields. 9, 9' - doublet-doublet-triplet signals of protons in symmetric carbon atoms are 3.28 m.u. observed in the fields. 11, 11' - the singlet signal of a proton in symmetric carbon atoms is 1.61 m.u. observed in the field (Table 1)

**Table 1.** $^{1}H$ and $^{13}C$ NMR chemical shifts of the compound (CDCl3, δ, ppm, 600 MHz) and HMBC experiment data

| Atom C | $\delta_C$ | $\delta_H$ (J/Hz) | HMBC (H→C) |
|---|---|---|---|
| 1, 1’ | 143.49 | | |
| 2, 2’ | 127.67 | 7.11, d (8.9) | 6, 6`, 4, 4`, 12 |
| 3, 3’ | 113.93 | 6.80, d (8.9) | 5, 5`, 1, 1`, 4, 4` |
| 4, 4’ | 156.25 | | |
| 5, 5’ | 113.93 | 6.80, d (8.9) | 3, 3`, 1, 1`, 4, 4` |
| 6, 6’ | 127.67 | 7.11, d (8.9) | 2, 2`, 4, 4`, 12 |
| 7, 7’ | - | - | - |
| 8, 8’ | 68.67 | 3.87, dd (11.1, 5.7) | 4, 9, 10, 4`, 9`, 10` |
| | | 4.15, dd (11.1, 3.0) | 4, 9, 10, 4`, 9`, 10` |
| 9, 9’ | 50.07 | 3.28, dd т (5.7, 4.1, | 8, 8` |
| 10, 10’ | 44.53 | 2.9) | 8, 9, 8`, 9` |
| - | - | 2.68, dd (5.0, 2.8) | 8, 9, 8`, 9` |
| 11, 11’ | 30.94 | 2.82, dd (5.0, 4.1) | 1, 1`, 12 |
| 12 | 41.59 | 1.61, c | - |

**Fig. 4.** Initial state of sunflower oil (a) epoxidized state of sunflower oil (b).

**3.4.The iodine number of epoxidized sunflower oil was determined**. The results are presented in Table 4. The iodine number (x) was calculated according to the following formula: X = ((A-V),75:100)/(0.1:1000) (1.1). Here, A is the volume of the alcohol control solution of iodine used for titration; ml. B - the volume of an alcohol solution of iodine used for experimental titration; ml. 0.75 g - a mass of iodine corresponding to 200 ml of 0.05% alcohol solution of iodine; 100 - recalculation to 100 g of oil; 1.3 - the weight of oil in grams; Conversion factor of 1000 mg of iodine to grams.

**Table 2.** Determination of iodine number of epoxidized sunflower oil

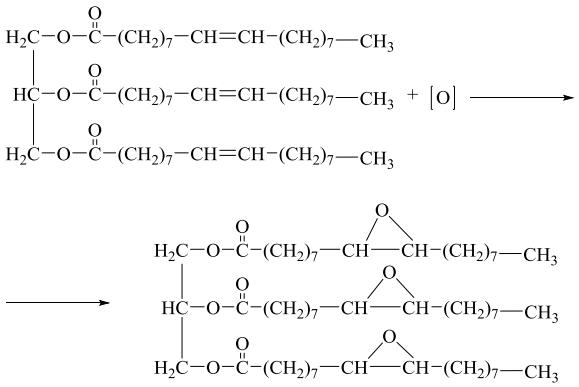
| System components and conditions | Number of iodine |
|---|---|
| Sunflower oil | 124,6 |
| Oil + hydrogen peroxide + formic acid + sodium bicarbonate (aqueous solution) + carbon dioxide gas 3h, 70°C | 117,8 |
| Oil + hydrogen peroxide + formic acid + sodium bicarbonate (aqueous solution) + carbon dioxide gas + tetramethyl ammonium bromide 24h, 140-150°C | 117 |

From Table 2, we can see that when sunflower oil is studied as a sample, its iodine number is equal to 124.6, and when it is processed, these indicators decrease. When vegetable oil was treated with hydrogen peroxide, formic acid, an aqueous solution of sodium hydrogen carbonate, and carbonic anhydride, it was found that the number of iodine in it changed to 117.8. When the effect of the catalyst on processed vegetable oil was studied, it was found that the number of iodine in it is equal to 117. As a result of the conducted scientific research, it can be said that after processing the vegetable oil, its iodine content was reduced. The method of adding hydrogen peroxide has a significant effect. Because hydrogen peroxide decomposes after 3 hours of practical experience. If an additional portion of hydrogen peroxide was added to the reaction mixture after 3 h, this resulted in a significant increase in product yield.

**Table 3.** Study of epoxidation process of vegetable oil (reaction time 6 hours at 60°C).

| Reagent (ml) | | | | Epoxidation yield, % |
|---|---|---|---|---|
| Amount of oil, ml | Formic acid, ml | $H_2O_2$, ml 30% | Additionally | |
| 46 | 7 | 15 | Triethanolamine titanate +0.2-ml $CO_2$ gas | 35 |
| 46 | 7 | 15 | Polybutyl titanate +0.3-0.4 ml of $CO_2$ gas | 32 |
| 46 | 7 | 15 | Tetrabutyl titanate+0.2 ml $H_2O_2$, 4gr $CO_2$ gas | 72 |
| 46 | 7 | 15 | 04 gr $H_2O_2$, Magnetic stirrer | 56 |
| 46 | 7 | 15 | Tetrabutylammonium bromide 0.2 ml $CO_2$ gas | 34 |
| 46 | 7 | 15 | Dichloroethane 0.1ml Magnetic stirrer | 65 |

Table 3 shows that the highest epoxidation yield of 72% was achieved when 0.2 ml of tetra butyl titanate was used as a catalyst against 46 g of oil, 4 g of hydrogen peroxide was added, and $CO_2$ gas was continuously passed. Also, substances such as triethanolamine titanate, poly butyl titanate, tetrabutylammonium bromide, and dichloroethane were used as catalysts. When these catalysts were used, the epoxidation yield was relatively low. Thus, research has shown that it is the most effective among the most environmentally friendly and inexpensive methods of epoxidation. According to the results of the conducted research, the epoxidation reaction was proposed as follows.



## Conclusion

As a result of the research, it can be concluded that the double bonds in the vegetable oil molecule form an epoxide group under the influence of oxidizing agents and with the presence of a catalyst. Hydrogen peroxide is the most effective of all epoxidizing agents available today. Hydrogen peroxide is cheap, non-toxic, and environmentally safe because it breaks down very easily into water and oxygen. Since the decomposition of hydrogen peroxide is faster than the epoxidation reaction of the compound, the hydrogen peroxide added to the epoxidation process should be added in several parts. The epoxidation of the compound also depends on the nature of the catalyst, and quaternary derivatives of titanium formed with organic radicals are effective for epoxidation reactions

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# ЭПОКСИДИРОВАННЫЕ ПОДСОЛНЕЧНЫЕ МАСЛА И ИХ ИССЛЕДОВАНИЕ

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**Аннотация.** В данном исследовании изучается эпоксидирование подсолнечного масла. Подсолнечное масло окисляли перекисью водорода в присутствии муравьиной кислоты. Процесс проводили в колбе, оснащенной смесителем и термометром. Подсолнечное масло окисляли 30%-ной перекисью водорода в присутствии муравьиной кислоты. В качестве катализатора использовали тетрабутилтитанат. Для удаления избытка муравьиной кислоты и перекиси водорода после завершения реакции реакционную смесь промывали раствором карбоната натрия и отделяли от водного раствора с помощью разделительной воронки. Затем его несколько раз промывали дистиллированной водой и сушили в вакууме. Состав и структура полученного эпоксидированного подсолнечного масла были изучены и проанализированы с использованием методов современной IQ-спектроскопии, рамановской спектроскопии, ЯМР и ПМР. Было также обнаружено, что он имеет йодное число и эпоксидное число. При IQ-спектроскопическом анализе образование эпоксидной группы было продемонстрировано наличием флуктуаций в области 2966,52-3055,24 $см^{-1}$. Подсолнечное масло имеет йодное число 124,6, и когда его окислили перекисью водорода с оксидом, было обнаружено, что количество йода в нем изменилось на 117,8.

**Ключевые слова:** подсолнечное масло, эпоксидирование, муравьиная кислота, перекись водорода, ИК-спектр, спектр комбинационного рассеяния света, ЯМР и ПМР.