

Vegetable Oils Epoxidation Mechanisms

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Abstract This article is a contribution on vegetable oils epoxidation mechanisms understanding which is of great interest in these last five years. Vegetable oils epoxidation mechanism is a chemical reaction that takes place at the double bonds. Most of the time, fats such as: myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, alpha linoleic acid and arachidic acid are found in these oils. However, temperature, oxygen and light are among the factors that destroy oils. Two methods are used for the epoxidation of the later: - Prilezhaev epoxidation which consists in the in situ formation of an organic per acid such as performic acid or peracetic acid. The latter is obtained by the action of hydrogen peroxide on the carboxylic acid in the presence of sulfuric acid; - The use of enzymes is a process for the elimination of sulfuric and the decreasing of the amount of secondary reactions. The use of enzymes gives a better selectivity on soybean and linseed oils, for example, respectively 94 and 98% with an exceptional conversion rate higher than 98% and showing almost no side reactions in this enzymatic epoxidation process. However, if the pH is very low or the temperature is higher than 60°C, the formed epoxy rings can react with the peracid and open up into hydroxyl and acetoxy groups. In addition, solvents such as dimethylformamide as well as chlorinated solvents are increasingly being excluded in industry for environmental reasons. Epoxidation with hydrogen peroxide is less demanding in terms of pH, temperature and solvent use. Moreover, this epoxidation method is not subject to side reactions. The peak characteristics of the epoxy function by Fourier Transform Infrared is located around 822cm⁻¹.

Keywords: vegetable oil, epoxidation, oxirane, environment

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1. Introduction

An oil is a fatty substance which is liquid at ambient temperature and immiscible with water. Oils are viscous and flammable. They have different origins: animal, vegetable, mineral. The parameters that differentiate oils in many cases and affect their physicochemical properties are the number of carbons that make up the fatty acids, the degree of unsaturation and the stereochemistry of the double bonds.

However, the aim of this article (not in an exhaustive way) is to conduct a global review study on the epoxidation of in the last five years, in order to have enough key information's from different sources and to reproduce them in a single document to facilitate the research on the subject of oils epoxidation. This is because there is no known studies in the domain of research in this context. This will also be our contribution.

2. The Chemical Nature of Vegetable Oils

Vegetable oils are triglycerides (a combination of three fatty acids and one glycerol molecule) whose composition depends on the nature of the plant, its growing conditions, the soil and the season. They are extracted from the plants seeds such as neem, jatropha, cotton, peanut, sunflower, soybean, etc. Oils have been part of human culture for centuries. Depending on their degree of saturation or unsaturation, they can be in liquid or solid form at room temperature. Their chemical structures are in the form of triglycerides, in which the three hydroxyl groups of glycerol are esterified by the fatty acid.

Fatty acids are carboxylic acids with very long aliphatic chains, saturated or not. The most found in oils are: myristic acid (C14-0), palmitic acid (C16-0), stearic acid (C18-0), oleic acid (C18-1), linoleic acid (C18-2), alpha linoleic acid (C18-3) and arachidic acid (C20-0).

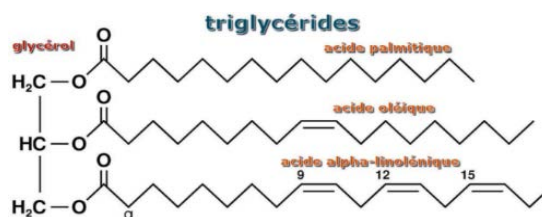


Figure 1. Chemical Structure of a triglyceride [1]

3. Vegetable Oils Composition in Terms of Fatty Acids Percentage

We present in Table 1 the fatty acid composition of the oils and the fatty acid molar percentage of some oils in Table 2.

Table 1. Fatty acids composition of vegetable oils

FAs [%]	SAF	GRP	SIL	HMP	SFL	WHG	PMS	SES	RB	ALM	RPS	PNT	OL	COC
C6:0	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.52
C8:0	nd	0.01	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	7.6
C10:0	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.01	nd	nd	5.5
C12:0	nd	0.01	0.01	nd	0.02	0.07	nd	nd	nd	0.09	nd	nd	nd	74.7
C14:0	0.10	0.05	0.09	0.07	0.09	nd	0.17	nd	0.39	0.07	nd	0.04	nd	19.9
C15:0	nd	0.01	0.02	nd	nd	0.04	nd	nd	nd	nd	0.02	nd	nd	nd
C16:0	6.7	6.6	7.9	6.4	6.2	17.4	13.1	9.7	20.0	6.8	4.6	7.5	16.5	nd
C17:0	0.04	0.06	0.06	0.05	0.02	0.03	0.13	nd	nd	0.05	0.04	0.07	nd	nd
C18:0	2.4	3.5	4.5	2.6	2.8	0.7	5.7	6.5	2.1	2.3	1.7	2.1	2.3	2.7
C20:0	nd	0.16	2.6	nd	0.21	nd	0.47	0.63	nd	0.09	nd	1.01	0.43	nd
C22:0	nd	nd	nd	nd	nd	nd	nd	0.14	nd	nd	nd	nd	0.15	nd
C16:1 (n-7)	0.08	0.08	0.05	0.11	0.12	0.21	0.12	0.11	0.19	0.53	0.21	0.07	1.8	nd
C17:1 (n-7)	nd	nd	0.03	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
C18:1 cis (n-9)	11.5	14.3	20.4	11.5	28.0	12.7	24.9	41.5	42.7	67.2	63.3	71.1	66.4	6.2
C18:1 trans (n-9)	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.14	nd	nd	nd
C20:1 (n-9)	nd	0.40	0.15	16.5	0.18	7.91	1.08	0.32	1.11	0.16	9.1	nd	0.3	nd
C18:2 cis (n-6)	79.0	74.7	63.3	59.4	62.2	59.7	54.2	40.9	33.1	22.8	19.6	18.2	16.4	1.6
C18:3 (n-3)	0.15	0.15	0.88	0.36	0.16	1.2	0.12	0.21	0.45	nd	1.2	nd	1.6	nd
C18:3 (n-6)	nd	nd	nd	3.0	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
SFAs	9.3	10.4	15.1	9.2	9.4	18.2	19.6	16.9	22.5	9.3	6.3	10.7	19.4	92.1
MUFAs	11.6	14.8	20.7	28.1	28.3	20.9	26.1	42.0	44.0	67.9	72.8	71.1	68.2	6.2
PUFAs	79.1	74.9	64.2	62.8	62.4	61.0	54.3	41.2	33.6	22.8	20.9	18.2	18.218.0	1.6
n-3 PUFAs	0.2	0.2	0.9	0.4	0.2	1.2	0.1	0.2	0.5	0.0	1.2	0.0	1.6	0.0
n-6 PUFAs	79.0	74.7	63.3	62.4	62.2	59.7	54.2	40.9	33.1	22.8	19.6	18.2	16.4	1.6

Data are expressed as percentages of total fatty acid methyl esters (FAMES); nd means that FAs was not determined Abbreviations of the sample mean SAF-safflower, GRP-grape, SIL-silybum marianum, HMP-hemp, SFL-sunflower, WHG-wheat germ, PMS-pumpkin seed, SES-sesame, RB-rice bran, ALM-almond, RPS-rapeseed, PNT-peanut, OL-olive, and COC-coconut oils.

Table 2. Vegetable oils composition in terms of fatty acid molar percentages [2]

Oil	C14-0(%)	C16-0(%)	C18-0(%)	C18-1(%)	C18-2(%)	C18-3(%)	C20-0(%)	Others (%)
Palm	0.5-2	32-45	2-7	38-52	5-11
Palm Kernel	14-18	7-9	1-3	11-19	0.5-2
Soy	<0.5	7-11	2-6	22-34	43-56	5-11
Sunflower	3-6	1-3	14-35	44-75	0.6-4	Behenic 0.8
Peanut	6-9	3-6	52-60	13-27	2-4	Behenic 1-3 Lignoceric 1-3
Cotton	0.4	20	2	35	42
Olive	0.1-1.2	7-16	1-3	65-80	4-10	0.1-0.3
Linen	4-7	2-5	12-34	17-24	35-60	0.3-1

In this table, we can notice that there only the presence of these 3 groups C18-1, C18-2 and C18-3. These three fatty acids correspond to soybean and flax seed oils with high molar percentages of fatty acids between 22 and 60%. These oils are major of interest for epoxidation because of their unsaturations. It is also noted in this table that these oils all contain at least two unsaturations whose percentage of fatty acids ranges between 0.5 and 80%. They also give a good selectivity in the case of epoxidation with enzyme also [3].

As the number of unsaturations increases, the iodine value increases. This is observed in the works of Rahman et al (2017). This makes these two to have higher iodine indexes than palm, olive, peanut, rape seed, sesame, cotton and wheat oils according to the composition of vegetable oils and their iodine value [3,4].

4. Oil Quality

To understand the nature of oils for epoxidation purpose, it is important to study some parameters.

4.1. Iodine Value

The iodine value (InI) is defined as the number of grams of iodine per 100g of fat. It was determined according to Wij's method. According to the experimental protocol used, an excess of iodine chloride is added to the fat in chloroform solution. After a few minutes of reaction, potassium iodide and distilled water are added. The liberated iodine is titrated by a solution of sodium thiosulfate (0.1 N) in the presence of starchpaste [5].

4.2. Saponification Value

The saponification index (InS) is defined as the amount of potassium hydroxide (KOH) in milligrams required to saponify one gram of fat. This parameter was determined according to the protocol described by the standard NF T 60-206 (AFNOR, 1984) [5].

4.3. Peroxide Value

The peroxide index of a fatty substance is the number of milliequivalents of active oxygen contained in a kilogram of product which oxidize potassium iodide with a release of iodine which amount is determined by sodium thiosulfate as a titration solution. It was determined according to the protocol described by the standard NF T 60-220 (AFNOR, 1984). The principle stipulates that the fat, in solution in acetic acid and chloroform, is treated with a solution of potassium iodide. The released iodine is titrated by a solution of sodium thiosulfate [5].

4.4. Acidic index Value

The acidic index (InA) is the number of milligrams of potassium hydroxide (KOH) required to neutralize the free fatty acids contained in one gram of fat; while the acidity of a fat is the percentage of free fatty acids conventionally expressed as oleic acid [6]. The acidity is deduced from the acid number and is a conventional expression of the

percentage of free fatty acids. These two chemical indicators were determined according to the standard NFV 03-906 (AFNOR, 1984) [5].

4.5. Calorific Capacity

The calorific (PC) capacity was calculated by using the following formula [5]:

$$PC = 47645 - 4,187 \text{ InI} - 38,31 \text{ InS} \text{ (kJ / kg)}$$

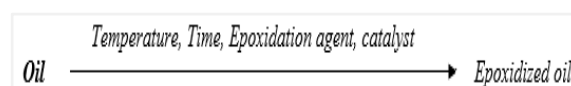
5. Vegetable Oil Epoxidation

The oil epoxidation is performed on the unsaturations of fatty substances. Moreover, it is one of the most important reactions for the functionalization of double bonds. It is conducted on a large scale in industry with an estimated 279.4 kilotons in 2020 of epoxidized soybean oil whose value in the global market is around 0.3 billion USD [2].

Vegetable Oils Epoxidized (VOE) are used as plasticizers and stabilizers for polyvinyl chloride and other plastics. These become flexible and sufficient to be used for the production of packaging films. They meet the requirements for high temperature lubrication and are easily biodegradable. A high reactivity of the oxirane ring in the oils causes their transformation into alcohols, polyols, glycol mono esters, glycol diesters, carbonyl compound and epoxy polymers.

5.1. Epoxidation Principle

It consists in reacting the oil and the epoxidizing agent such as hydrogen peroxide (H_2O_2) in the presence of a catalyst such as formic acid or sulfuric acid.



Scheme 1. Principle of epoxidation reaction

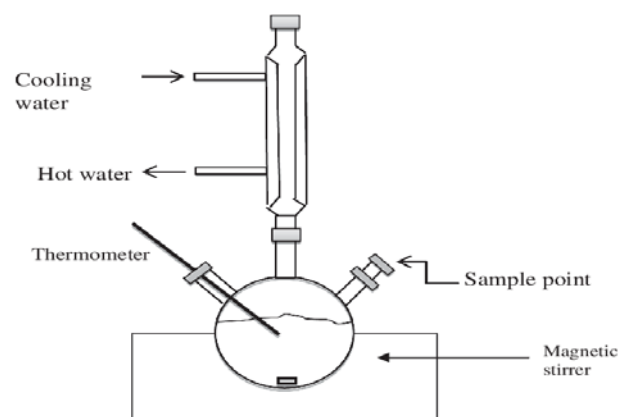


Figure 2. Expérimental setup for oils epoxidation

5.2. Factors that Make Oils Ranced

Oils are sensitive to certain natural factors which, depending on where they are stored, can denature them quite easily:

- Oxygen O₂: the presence of oxygen promotes rapid oxidation. Some manufacturers replace the air remaining in the bottle by nitrogen to allow the oil to keep as long as possible. DRIDI Wafa et al [7] have detailed the effect of oxygen on oils oxidation.
- UV rays: UV rays are catalysts that can amplify the oxidation reaction. It is there for synonymous with an acceleration of the oxidation reaction in the oil is exposed to the sun.
- Heat: it destabilizes the double bond C=C and can initiate the oxidation reaction by catalyzing it. Thus, to benefit from the nutritional qualities of an oil, it is important to choose a cold extracted oil. In general, an increase in temperature leads to an increase in the rate of oxidation which follows the Arrhenius law [7].

5.3. How to Choose Oxidizing Agents and Solvents

5.3.1. Oxidizing Agents

Epoxidation reactions require a good choice of the oxidizing agent. This is always based on the reactivity, the percentage of active oxygen as well as the product obtained during the epoxidation reaction and the by-product generated [8].

Table 3. Oxidizing agents and their percentages of active oxygen for epoxidation reactions

Oxidant	Active oxygen (%)	Sub-product generated
O ₂	100	None
O ₂ (reduction agent)	50	H ₂ O
H ₂ O ₂	47	H ₂ O
NaOCl	21,6	NaCl
TBHP	17,8	Tert-butanol
KHSO ₅	10,5	KHSO ₄
m-CPBA	9,3	Chlorobenzoicacid
PhIO	7,3	PhI

Luc Charbonneau [8] showed the importance of the choice of each of the oxidants in the case of the epoxidation of olefins. These oxidants are also widely used in the epoxidation of oils such as H₂O₂.

5.3.2. Solvents

Whether in homogeneous or heterogeneous conditions, the epoxidation reaction generally requires the presence of a polar solvent. Due to human health and new environmental measures, several solvents such as polar solvents except alcohols, esters (ethyl or propylacetate), acetonitrile or ketones (acetone or methylethylketone), dimethylsulfoxide (DMSO) and dimethylformamide (DMF) as well as chlorinated solvents are excluded in industry [8].

Therefore, protic solvents can cause the opening of the oxirane ring forming a glycol ether [8].

5.3.3. Use of Catalysts

The use of catalysts in the case of oil epoxidation such as enzymes, ion exchange resins [9,10] acid, metal

catalysts, inorganic acids, is widely studied explicitly by JUN LIN ZHENG et al [2].

5.3.4. Oil Clarification

Oils may contain impurities after their extraction. For this reason, they should be treated to avoid any disturbance during the chemical reaction and impact on the product. Thus, there are semi-artisanal, artisanal [11] and chemical clarification. The latter is done either by single or multiple clarification:

• Unique clarification

The vegetable oil is filtered to remove solid impurities before being diluted in dichloromethane (CH₂Cl₂) to reduce the viscosity. This solution is washed with a NaCl solution at 60°C. Thus, there will be the presence of two phases of which the organic phase is dried on MgSO₄ [12] then filtered and the solvent is removed using a rotary evaporator. As a result, a clear brown oil is obtained.

• Multiple clarification

Similarly, the vegetable oil is filtered to remove solid impurities and then diluted in CH₂Cl₂ to reduce viscosity. The mixture is washed with a saturated NaCl solution at 60°C. However, the mixture is allowed to stand until phase separation, the organic phase is collected and the procedure is repeated more than four times. The crude oil dried on MgSO₄ [12], is filtered to remove the drying agent. And the solvent is then removed using a rotary evaporator to obtain a clear brown oil.

6. Experimental Protocols

The experimental protocol for epoxidation of oils has been described in the literature. This is the case of Jun Liu Zhen et al. [2] who worked on the epoxidation of cotton seed oil.

They [Jun Liu Zhen et al. [2] introduce in a 500 mL reactor where the mixture is stabilized at 60°C for quantities of 180 g of the vegetable oil, 180 g of hydrogen peroxide (33 wt%) and 50 g of water. Thus, formic acid is added to the reaction with a flow rate of 2.9 mL/min for 25 minutes. The stirring is stopped after one hour of reaction to allow the separation of the two phases. Therefore, the organic phase is washed once with 300 mL of a 10 wt% aqueous sodium carbonate solution, then three times with 200 mL of water. The product is dried in a rotary evaporator and then over anhydrous magnesium sulfate. The final product is stored at 3°C under argon once filtered to avoid oxygenation. They obtained a conversion of 97% of this oil with a selectivity of 81% determined by titration.

Other epoxidation protocols are described in the literature [4,12-24].

7. Epoxidation Mechanisms

7.1. Prilezhaev's Method

This is the most used method in the industry called the Prilezhaev epoxidation. This process consists of the in situ formation of an organic peracid such as performic acid or peracetic. The latter is obtained by the action of

hydrogen peroxide on the carboxylic acid in the presence of H_2SO_4 reaction a) diagram 2. Guillaume Falco and al (2016) [3] have shown the disadvantages of this process, namely:

- The presence of H_2SO_4 leads the reaction to low selectivity rarely beyond 80%.
- Difficult separation of products at an additional neutralization stage and risks of corrosion of the chemical equipment used an ion exchange resin can replace the acid in order to secure the process
- The use of a peracid can lead to side reactions and more particularly the opening of the epoxy cycles formed, increasing the viscosity of the epoxidized vegetable oil. They conclude, despite the partial consumption of epoxy rings during secondary reactions, the conversion of double bonds reaching 90%. Guillaume Falco and al (2016) [3] also claim that the use of peracetic acid compared to performic acid gives a low number of ring openings.

7.2. Enzymatic Way

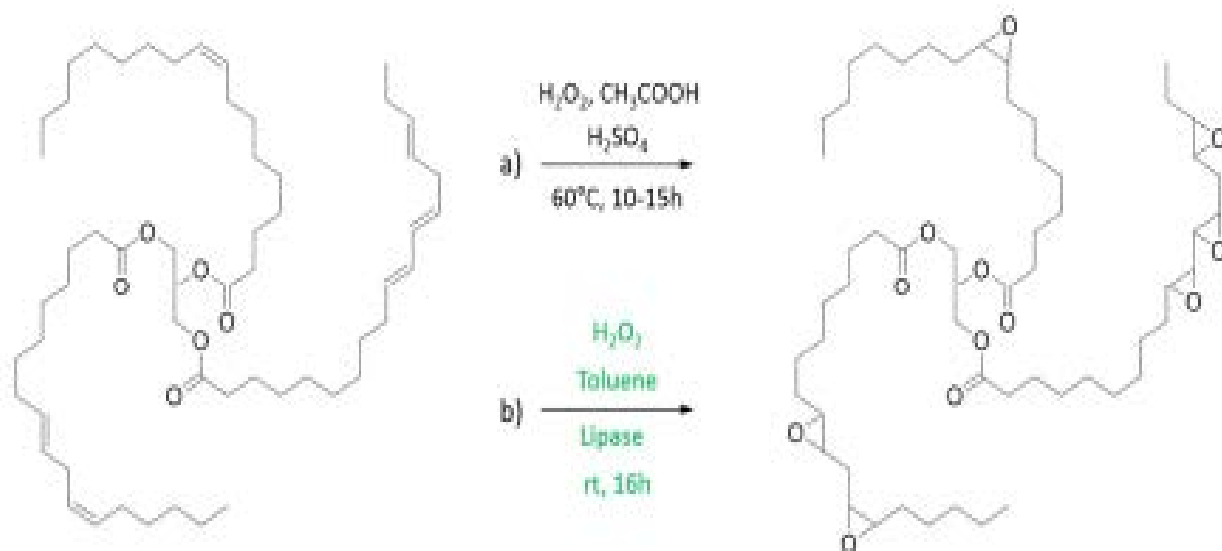
This enzymatic process was used to eliminate the strong acid H_2SO_4 and to decrease the percentage of side

reactions. This process is less hazardous and more respectful for the environment. This method was studied using a hydrolysis process catalyzed by a lipase reaction b) Scheme 2.

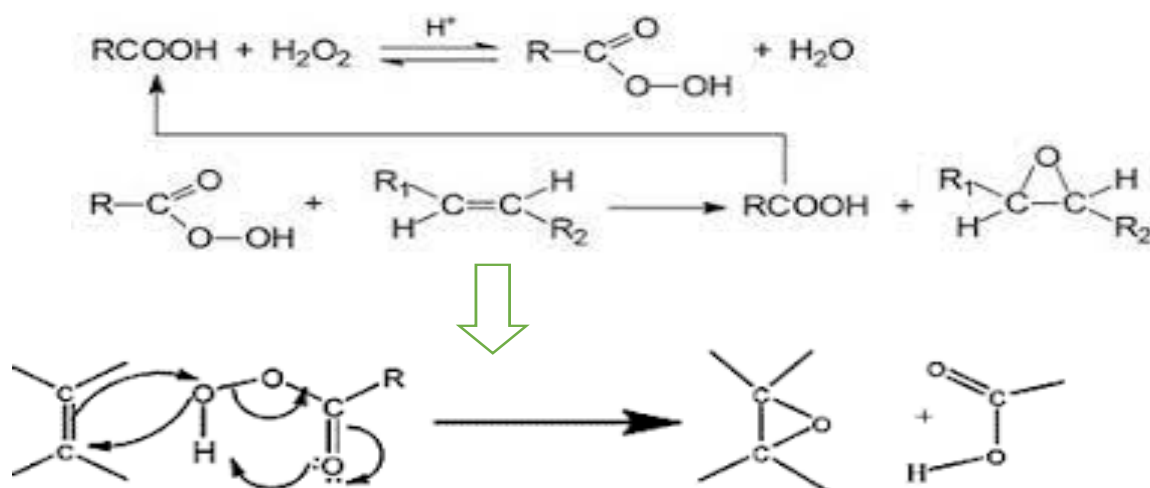
According to Guillaume Falco et al (2016) [3], the use of enzymes gives a better selectivity on soybean and linseed oils respectively 94 and 98%. They obtained an exceptional conversion rate greater than 98% and also showing almost non-existing side reactions in this enzymatic epoxidation process.

The epoxidation of peracids is the most widely used method in industry. In this case, the epoxidation reaction takes place in acetic acid solution to avoid side reactions. This reaction requires good control of the operating conditions to obtain desired yields.

However, if the pH is very low or the temperature exceeds $60^\circ C$, the formed epoxy rings can react with the peracid and open into hydroxyl and acetoxy groups. Epoxidation with hydrogen peroxide is less restrictive in terms of pH, temperature and the use of solvents. In addition, this epoxidation method is not subject to side reactions. On the other hand, it requires the use of a phase transfer catalyst in order to obtain good yields.



Scheme 2. Epoxidation methods of oils: a) the Prilezhaev epoxidation; b) perhydrolysis catalyzed by lipase-type enzymes



Scheme 3. Mechanism of epoxidation of oils with peroxide [14]

8. Determination of the Rate Relating to the Conversion to Oxirane

The calculation of the rate relating to the conversion into oxirane is defined by some authors such as Eni Budiayati and al (2020) [15], Canan TAS and al (2018) [13], Marlana Music and al (2018) [14], Fernandes and al (2017) [12], Shotori S.Meadows and al (2017) [16], Anand V. Parwardhan and al (2016) [17], Dinda. S and al (2016), Arnis Abolins and al (2020) [9]. Marlana Musikand al (2018, 2017) [17] established the conversion formula and the expression allowing the calculation of selectivity.

9. Kinetic Study and Thermodynamic Parameters of the Epoxidation Reaction

The kinetic and thermodynamic study will make it possible to understand the course of the epoxidation reaction based on parameters such as speed, time, concentrations of reactions and products, enthalpy, entropy, activation energy etc. Several studies have been conducted in this context by [25-30].

10. Characterization of the Oxirane Group by Fourier Transform Infrared Spectroscopy (FTIR)

Shotori S. Meadows and al had to work in 2017 [16] on the epoxydation of soybean oil. In order to understand the epoxide group formation, they referred to ASTM D1652-11 standard to perform FTIR spectroscopy. The peak at 822 cm^{-1} verifies the presence of epoxy groups indicating that double bonds exist and decrease with increasing molar hydrogen peroxide as indicated by ASTM D1652-11. This same peak is found in the FTIR characterization of soybean oil by Arnis Abolins and al (2020) [9] and Halleux and al (2019) [25]. Figure 2 and Figure 3 [31] give a more in-depth view of the formation of epoxy groups at 822 cm^{-1} which corresponds to the data obtained from the standard ASTM D1652-11 [31].

Moreover, the result of the absence of an epoxide peak justifies the introduction of secondary reactions and the appearance of an oxirane cleavage which degrades the epoxide rings. Other peaks observed by Shotori S. Meadows and al (2017) [16] from the FTIR spectrum are 3530 cm^{-1} (OH stretch), 3012 cm^{-1} (C-H and C=C stretch), 2920 cm^{-1} (C-H stretch of CH_3), 2850 cm^{-1} (C-H stretch CH_2 stretch), and 1738 cm^{-1} (C=O carbonyl ester stretch vibration). The peak at 3530 cm^{-1} corresponds to the secondary reactions which disturb the oxygen content of the oxirane due to the OH groups. Verification of this result is expressed by the peak at 3530 cm^{-1} . The peak at 3012 cm^{-1} corresponds to the level of unsaturation which disappears as the double bonds are converted and used, thus correlating with the epoxide peak at 822 cm^{-1} [31].

11. Epoxy Market Research

The global epoxy resin market size is expected to reach \$ 19.55 billion by 2028 [31]. It is projected at a CAGR (growth rate of one compound per year) of 6.7% over the forecast period. Thus, in the oil and gas industry the market is expected to witness an impressive CAGR of 6% over the next five years, driven by an expected increase in oil and gas platform business, increasing the demand for non-corrosive and light weight composites, and continued investment in pipeline infrastructure to meet growing demand from developing countries due to population growth [31]. Therefore, COVID-19 has impacted oil and gas epoxy resin market dynamics, competition and global supply chain [32] gradually an upward trend from 2021 Figure 3. For the search for renewable resources, studies on epoxy resins are constantly increasing. Indeed, we are witnessing an increase in the number of publications of the last four years Figure 4 [33].

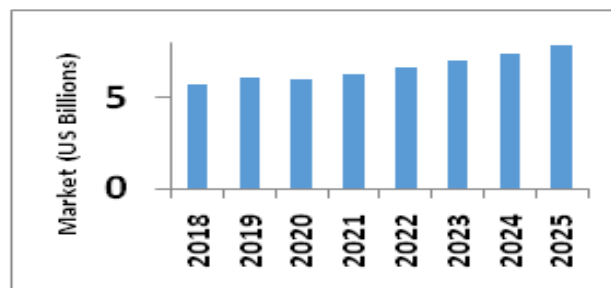


Figure 3. Impact of Covid'19 on the dynamics

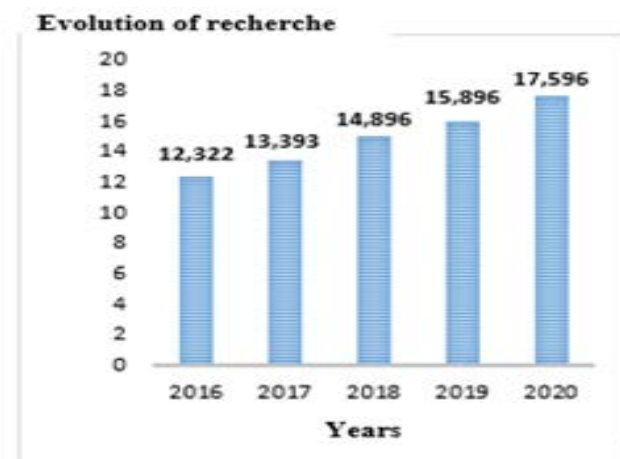


Figure 4. Evolution of epoxy research per year

Global epoxy coatings market was evolved at USD 27.66 billion in 2017 and is projected to reach USD 38.92 billion by 2024, growing at a rate of 5% between 2018-2024 [34]. It is driven by various factors such as the increase in demand from developing countries. However, strong demand in end-use industries is estimated to drive the epoxy coatings market over the forecast period. Moreover, the non-VOC type of coatings, powder-based epoxy coatings are gaining popularity. The rapid growth of the coatings market is due to the development of new formulation, material and equipment. **Asia-Pacific** accounted for the largest market share in 2017 for epoxy coatings due to high demand from developing economies as well as developed countries. **China** was the largest

market in 2017 and is estimated to dominate in the near future [34]. The rapid growth of end-use industries which includes petrochemicals and steel have increased the demand for epoxy coatings in **China**, **India** and **Taiwan** are also showing substantial growth over the forecast period. Thus, **Indonesia** is considered as the hub of chip building, automotive and one of the end users of industries using epoxy coating [34]. The growing demand for green buildings in North **America** is the key sign for the increase in the epoxy coatings market in the region. Europe is the third largest market for epoxy coatings after **AsiaPacific** and North **American** [34]. **Europe** is expected to show significant rate growth over the projected period as it is a mature market compared to **AsiaPacific**, **Africa**, **Latin America** and the Middle East. This last, **Africa** and **Latin America** should be a potential market for epoxy coatings because it is a less developed region. Key players in epoxy coatings are AKZONOBEL NV, RPM International Inc, BASF SE, le Valspar Corporation, PPG Industries, Nippon Paint Co Ltd, Sherwin-Williams Company, Tikkurila OYJ, Axalta Coatings Systems LLC, and Kansai Paint Co Lt [34].

12. Conclusion

It is shown in this article the composition in mass percentage in fat of certain vegetable oils, the method of purification of fat oils, and the role of oxidants but also of the protocols of epoxidation of oils are keys parameters in vegetable oil epoxidation. This synthesis of scientific documents and publications from the last five years is far from being exhaustive but will provide necessary information's of the topic of oil epoxidation and the techniques for characterizing them. Thus, enzymatic epoxidation is of significant interest with satisfactory results. Hence the study has helped to understand the current state of functioning of the global epoxy market.

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