

OPTIMIZATION OF EPOXIDATION PALM-BASED OLEIC ACID TO
PRODUCE POLYOLS

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Abstract. Optimization of epoxidation by using response surface methodology (RSM) based on three-level three-factorial central composite design (CCD) was used. Response percentage of relative oxirane content (%RCO) was studied to determine the optimum reaction condition for production of polyols. The predicted value of model (85 %) was excellent in accordance to experimental value (81 %). All parameters (temperature, molar ratio of formic acid to oleic acid and molar ratio of hydrogen peroxide to oleic acid) were significant in influencing the course of epoxidation reaction ($p < 0.05$). The interaction between all parameters is also highly significant with $p < 0.0001$. Optimum reaction conditions obtained from RSM were as follows: the temperature 318 K, molar ratio of formic acid to oleic acid 1.64:1 and molar ratio of hydrogen peroxide to oleic acid 2:1. The epoxidation of palm oleic acid was carried out by using *in situ* performic acid. FTIR analysis showed the formation of epoxy functional groups at optimum reaction condition at the wavelength of 1340 cm^{-1} . This epoxide group was used to produce polyols by using hydroxylation process and the polyols functional group was detected at the wavelength of 816 cm^{-1} .

Keywords: temperature, molar ratio, formic acid, hydrogen peroxide, epoxidation, polyols.

1. Introduction

Epoxide is a cyclic ether with three-membered ring, containing two carbon atoms and one oxygen. There are many sources that can be used to produce epoxide such as soybean oil, sunflower oil, palm oil, *etc.*¹ There are two methods that can be used to produce epoxy groups such as epoxidation with halohydrins or molecular hydrogens and epoxidation with performic acid generated *in situ* or preformed.² Performic acid generated *in situ* has been selected as an epoxidation process in this study since it is low in price and eco-friendly. In terms of application, it is used as plasticizer, for stabilization in polyvinyl chloride and intermediates product such as for polyols production.³

Over the past decades, petrochemical-based raw material was used for polyols production. This raw material has economic and environmental challenges. The cost of its production is quite high. In addition, it also contributes to environmental problem such as air pollution. Currently, it has been notified that polyols also can be produced from vegetable oils like palm oil, soybean oil and rapeseed oil. Among these precursors, palm oil is one of the best oils to produce polyols because of availability of raw material, cost effectiveness and environmental friendliness.⁴ Polyols are an intermediate product vastly used in as pharmaceutical, food, polymer, and other industries. The major uses of polyols is in polymer industry where it is used in the production of polyurethanes (PU) such as foam, coating and elastomer.⁵ The common method of producing polyols are by transesterification of fatty acid, by hydroformylation, by oxidizing of olefin, epoxidation, and hydroxylation process from vegetables oil.⁶ In this study, epoxidation and hydroxylation methods were used to produce polyols due to high volume of production, fast reaction time, eco-friendliness, and low cost of production.⁷

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Response surface methodology (RSM) is an optimization tool to evaluate the relative significance of several factors in the presence of complex interactions. It is a powerful technique for testing multiple-process variables with low number of experimental trials. RSM is able to determine the optimum levels of applied factors in order to obtain the desired (smallest or largest) value of the response function.⁸ In this study, CCD-RSM was used to study the optimum reaction conditions for epoxidation process to achieve high percentage of relative conversion of oxirane content (% RCO). The parameters involved are the temperature, unsaturation molar ratio of formic acid to oleic acid and unsaturation molar ratio of hydrogen peroxide to oleic acid. The optimum reaction condition of epoxidized oleic acid was used for polyols formation.

2. Experimental

2.1. Materials

Palm-based oleic acid (75%) was used as a raw material; hydrogen peroxide (30%), formic acid (85%), sulfuric acid (95%), hydrogen bromide (48%), glacial acetic acid (100%), methanol and isopropanol (100%), and crystal violet were purchased from Qrec Sdn Bhd.

2.2. Epoxidation Process

Epoxidation process was carried out in a 250 mL beaker with agitation speed of 1000 rpm using magnetic stirrer. Oleic acid, formic acid or sulphuric acid were added as catalysts into a round bottom flask. The mixture was stirred continuously at fixed speed and gradually heated to required temperature and kept at constant temperature for the selected reaction time. The hydrogen peroxide was then added dropwise at desired temperature

under continuous stirring. The samples were analysed by withdrawing approximately 5 mL (4-5 g) of reaction mixture every 5min.

2.3. Preparation of Polyol

Hydroxylation process was carried out in a 250 mL beaker, similar to epoxidation process. 100 g of oleic acid was hydroxylated using a mixture of alcohols (methanol 25 g and isopropanol 25 g). The reaction was conducted at the temperature of 338 K, at 1000 rpm for 2 h. The reaction product (polyol) was analysed using FTIR to identify the presence of functional groups. The details of parameter involved and the reaction conditions of epoxidation process and polyols are given in Table 1. The experimental setup for epoxidation process and preparation of polyol can be seen in Fig. 1.

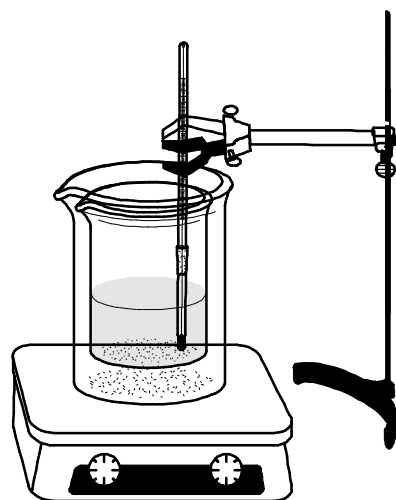


Fig. 1. Laboratory setup for epoxidation and polyols

Table 1. Experimental design for epoxidation and polyols

Reaction parameter	Range	Units
Epoxidation process		
Temperature of oleic acid and hydrogen peroxide	318–368	K
Formic acid to oleic acid ratio	1:2	-
Hydrogen peroxide to oleic acid ratio	1:2	-
Agitation speed	1000	Rpm
Reaction time	45	Minutes
Catalyst (sulphuric acid)	0.2–0.5	Gram
Formation of polyols		
Oleic acid epoxidation	100	Gram
Methanol	25	Gram
Isopropanol to oleic acid epoxidation	25	Gram
Operating temperature for polyols	338	K
Reaction time for polyols	2	hours

2.4. Determination of Experimental Oxirane Oxygen Content

The experimental oxirane oxygen content was determined by following American Oil Chemists's Society 1997 (AOCS) Tentative method Cd 9-57. 10 mL of acetic acid were added into an Erlenmeyer flask and the mixture was shaken. Next, two drops of crystal violet indicator were dropped into the mixture. Then, the mixture was titrated under stirring with hydrogen bromide until bluish green colour was obtained and left for at least 30 s.⁹ The equation to calculate (OOC_{exp}) in moles per 100 g is given as follows:

$$OOC_{exp} = \frac{T \cdot N_{HBR} \cdot 1.6}{W} \quad (1)$$

where T is a volume of hydrogen bromide required to titrate the sample, mL; N_{HBR} is a normality of the hydrogen bromide; W is weight of the sample, g.

From Eq. (1), the relative percentage conversion to oxirane content (%RCO) can be calculated through Eqs. (2) and (3).

$$RCO = \frac{OOC_{exp}}{OOC_{theor}} \cdot 100\% \quad (2)$$

$OOC_{theor} = (IV_o A / 2A_I) / (250 + (IV_o A / 2A_I) A_o) \cdot A_o \cdot 100$ (3) where OOC_{theor} is a theoretically maximum oxirane oxygen content, mg/L; IV_o is an initial iodine value of oleic acid sample; A_o and A_I are the molar masses of oxygen and iodine, respectively, kg/mol.

2.5. Response Surface Methodology

The three-level, three-factorial CCD experimental design with categorical factor was employed to study the effect of temperature, unsaturation molar ratio of formic acid to oleic acid, and unsaturation molar ratio of hydrogen peroxide to oleic acid based on the %RCO (response). The design was composed of three levels (low, medium, and high), being coded as -1, 0 and +1. The total of 20 runs was carried out to optimize the level of chosen variables. For statistical computation, these variables were denoted as A, B and C, respectively, as tabulated in Table 2.

Table 2. Codes and actual levels of independent variables

Symbols of independent variables	Coded levels		
	-1	0	+1
A (temperature, C)	45	70	95
B (unsaturation molar ratio of formic acid to oleic acid)	1.0	1.5	2.0
C (unsaturation molar ratio of hydrogen peroxide to oleic acid)	1.0	1.5	2.0

Table 3. Central composite design (CCD) and its corresponding values

Run order	Factor 1	Factor 2	Factor 3	Response	
	Temperature*	Molar ratio of formic acid to oleic acid	Molar ratio of hydrogen peroxide to oleic acid	% RCO (Actual)	% RCO (Predicted)
1	70	1.5	2.0	55.1249	55.6552
2	70	1.5	1.5	46.3768	47.6504
3	70	2.0	1.5	36.7183	36.9313
4	70	1.0	1.5	26.0480	25.0226
5	45	1.0	1.0	30.7840	28.4976
6	95	2.0	2.0	54.0000	52.0750
7	95	1.0	1.0	50.2700	49.8491
8	45	2.0	1.0	70.3297	70.2168
9	95	2.0	1.0	57.6577	57.6302
10	70	1.5	1.5	48.8596	47.6504
11	45	1.0	2.0	59.4657	59.1410
12	70	1.5	1.5	47.4047	47.6504
13	70	1.5	1.0	42.8380	43.1111
14	45	1.5	1.5	73.8817	73.1988
15	70	1.5	1.5	48.9297	47.6504
16	45	2.0	2.0	75.9794	75.177
17	70	1.5	1.5	47.4074	47.6504
18	95	1.5	1.5	71.8025	72.3234
19	95	1.0	2.0	70.8749	69.9768
20	70	1.5	1.5	46.7836	47.6504

Note: temperature is in Celsius degree for calculations

3. Results and Discussion

3.1. Statistical Analysis

3.1.1. RSM model development

In this research work, three operational parameter including temperature, unsaturation molar ratio formic acid to oleic acid and molar ratio of hydrogen peroxide to oleic acid have been chosen as operating variables and the percentage of RCO was the response. 20 sets of experiments were performed. The corresponding results of these experimental sets are summarized in Table 3.

Table 3 shows the different percentage of RCO obtained when varying level of parameters. Each of parameter affects the reaction condition and response. In this study, the highest percentage of RCO was 76 % at the run order of 16.

3.1.2. Analysis of variance

Analysis of variance (ANOVA) is a statistical method used to determine the differences among group means in the sample.^{9,10} The ANOVA results are tabulated in Table 4. The quadratic equation for predicting the optimum conditions was obtained from CCD-RSM and input variables. Thus, the empirical relationship between the response and independent variables is presented as Eq. (4):

$$Y = -56.95 - 4.486A + 284.60B + 40.97C + 0.040762A^2 - 63.93B^2 + 6.46 - 0.6555AB - 0.1738AC - 23.65BC \quad (4)$$

where Y is %RCO (predicted), A, B and C are temperature, molar ratio of formic acid, and molar ratio of hydrogen peroxide, respectively.

Table 4. Central composite design (CCD) and its corresponding values

Source	Sum of square	df	Mean square	F-value	p-value	Significant
Model	3773.92	9	419.32	597.84	0.000	Significant
Linear:	735.13	3	245.04	349.36	0.000	Significant
A	3.41	1	3.41	4.85	0.0052	Significant
B	327.67	1	327.67	467.16	0.000	Significant
C	404.06	1	404.06	576.07	0.000	Significant
Square:	2184.37	3	728.12	1038.10	0.000	Significant
A ²	1784.82	1	1784.82	2544.64	0.000	Significant
B ²	702.50	1	702.50	1001.56	0.000	Significant
C ²	7.18	1	7.18	10.23	0.000	Significant
Two-way interaction:	854.42	3	284.81	406.05	0.000	Significant
AB	537.04	1	537.04	765.67	0.000	Significant
AC	37.78	1	37.78	53.86	0.000	Significant
CC	279.60	1	279.60	398.62	0.000	Significant
Std. Deviation	0.837498					
R ²	0.9981					
Adjusted R ²	0.9965					
Predicted R ²	0.9946					

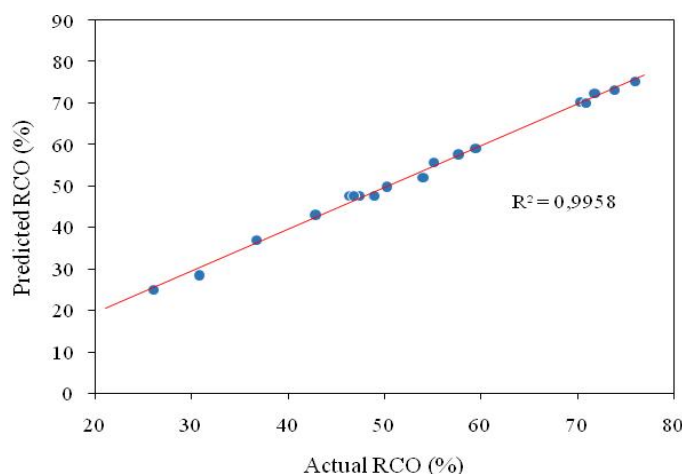


Fig. 2. Actual and predicted values for RCO percentage

The ANOVA results referring to equation and actual relationship between the response and significant variables represented by the equation are accurate. The significance of the coefficient term is determined by the values of F and p . The larger the value of F , the smaller the value of p , the more significant the coefficient.¹¹ The linear model is statistically significant if p -value is less than 0.05. Other than that, the square and two-way interactions model would be significance if p -value is less than 0.0001.¹² From the results obtained in Table 4, all the parameters in linear model are highly significance parameters with p -value less than 0.05. Among these control factors, parameter C (unsaturation molar ratio of hydrogen peroxide to oleic acid) is the most influential factor affecting the response. This is due to the largest number of F -value. These trends also work for square and two-way interaction models whereby all the parameters are regarded as highly significant because of p -value less than 0.0001.

Fig. 2 shows the graph of actual and predicted value for percentage of RCO. This graph was analysed to examine the correlation between the actual and predicted responses. From the results it can be observed that the data points were well distributed close to straight line indicating the best fitting between the actual and predicted values of the response. The value of R is the variability between the actual value and predicted value. In this study, the value of R^2 is 0.9958 which implies that 99.58 % of actual data agreed well with the predicted data.¹³

3.2. Interaction Effect Between Operating Parameters

Fig. 3 shows the interaction between temperature (A) and molar ratio of hydrogen peroxide (C) towards the

percentage of RCO. The optimization condition can be seen clearly at the contour plot graph. The smallest of %RCO is referring to the clearest colour with percentage less than 50 %. Under this condition, temperature range and molar ratio of hydrogen peroxide are approximately 329–356 K and 1.0–1.68, respectively. As reported by previous study,¹⁴ high operating temperature will contribute to low oxirane content and *vice versa*. This is because high temperature will promote the degradation of RCO.¹⁵ It is also highlighted that molar ratio of hydrogen peroxide is one of the crucial factors that contributes to %RCO. It shows that when molar ratio of hydrogen peroxide increased, the %RCO also improved progressively. However, the reaction becomes irreversible if the molar ratio content is too high.¹⁶ Referring to graph, the suitable operating temperature and molar ratio of hydrogen peroxide were 318 K and 2.0, respectively.

The effect of temperature and molar ratio of formic acid on %RCO at constant molar ratio of hydrogen peroxide is shown in Fig. 4. As can be seen in contour plot, more than 70 % of %RCO was achieved at 318 K of operating temperature and molar ratio of formic acid 1.64. As can be seen from this interaction, the smallest %RCO is less than 30 % within the range of operating temperature and molar ratio of formic acid of approximately 328–351 K and 1.0–1.03, respectively. According to the results obtained by Lee *et al.*¹⁷ and Derouet *et al.*,¹⁸ the highest molar ratio of formic acid results in higher formation of %RCO. On the other hand, the trend is totally different with temperature, since low temperature is favourable for the formation of %RCO.¹⁹ This temperature is vital to achieves high oxirane content. It is also proven by statistical analysis (ANOVA) that when the p -value is less than 0.05 these parameters are significant to the response.

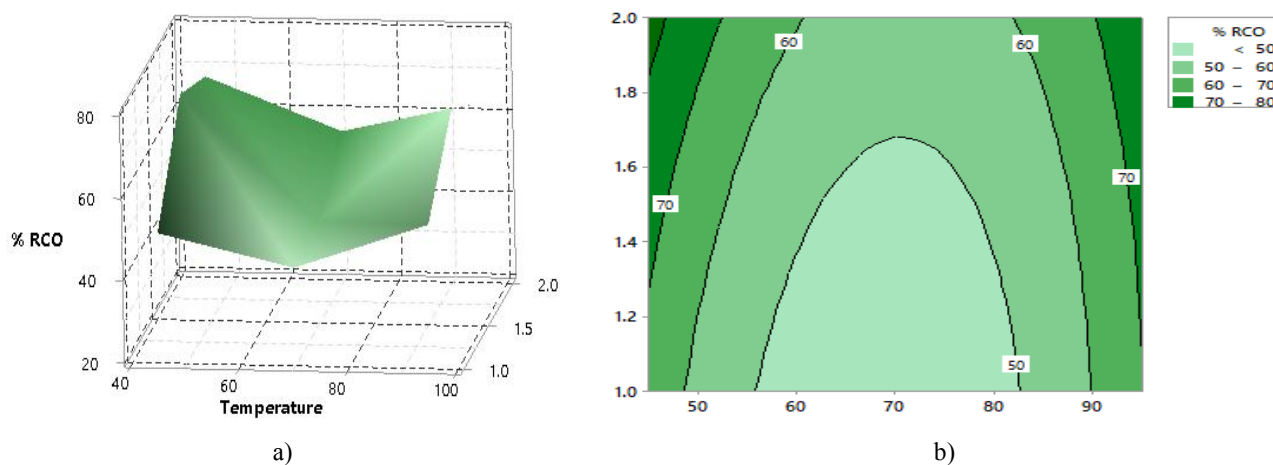


Fig. 3. Three-dimensional (a) and contour (b) plots for interaction between temperature and hydrogen peroxide towards % RCO

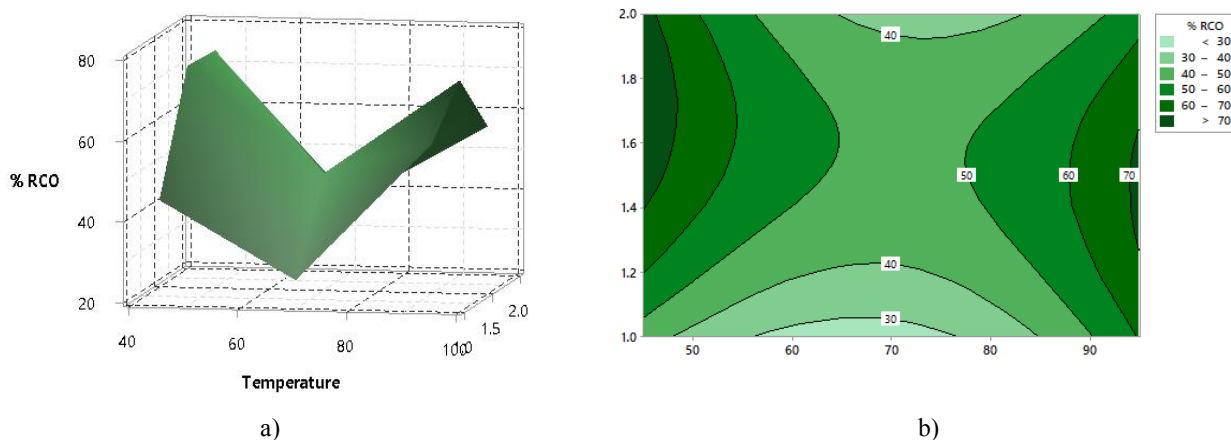


Fig. 4. Three-dimensional (a) and contour (b) plots for interaction between temperature and formic acid towards % RCO

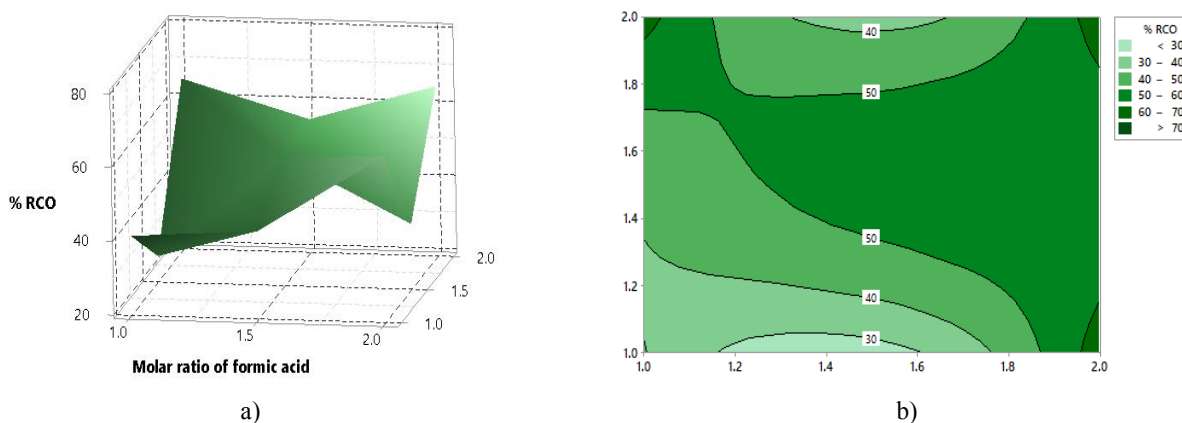


Fig. 5. Three-dimensional (a) and contour (b) plots for interaction between molar ratios of formic and hydrogen peroxide towards %RCO

Fig. 5 indicates the interaction between molar ratio of formic acid and hydrogen peroxide towards %RCO. Based on the graph, the suitable molar ratio for both parameters are in the range of 1.5–2.0 at 318 K reaction temperature to obtain the %RCO more than 70 %. Generally, higher molar ratio for hydrogen peroxide is pronounced to obtain high oxirane ring opening, which is slightly different with formic acid.²⁰ Unlike hydrogen peroxide, the molar ratio of formic acid should be less than 2.0 in order to prevent instability of oxirane ring. In addition, high molar ratio of formic acid can cause hydrolysis which tends to decrease %RCO.²¹

3.3. Optimum Reaction Conditions

Based on the reported results, optimisation of the %RCO parameters was carried out using a numerical optimisation method. The optimum reaction condition calculated on the %RCO for oleic acid epoxidation were operating temperature of 318 K and unsaturation molar ratio of formic acid and hydrogen peroxide of 1.64 and 2.0,

respectively. The amount of %RCO of predicted and experimental at optimum conditions is indicated in Table 5.

Table 5. Validation of optimum operational conditions

%RCO		
Predicted	Experimental	Errors
84.7229	80.5538	4.9

$$\% \text{ errors} = \frac{\text{predicted} - \text{experimental}}{\text{predicted}} \cdot 100 \quad (5)$$

$$\% \text{ errors} = \frac{84.7229 - 80.5538}{84.7229} \cdot 100 = 4.9\%$$

Comparing the experimental and predicted results it can be seen that the error between the experimental and predicted values is about 4.9 % (Eq. 5). Therefore, it can be concluded that generated model has sufficient accuracy to predict the amount of %RCO. The optimum conditions underwent confirmation test in order to study the sole effect of temperature, unsaturation of formic acid to oleic

acid and molar ratio of hydrogen peroxide to oleic acid toward %RCO.

3.5. Validation Test

The properties of each chemical were examined by Fourier Transform Infrared (FTIR) spectroscopy. FTIR was used to determine the C=C double bonds in the oleic acid and the formation of an oxirane ring in epoxidized oleic acid and to identify the formation of alcohol groups (OH groups), long chain hydrocarbon groups and the

opening oxirane content in polyol. The FTIR spectrum for these chemicals is shown in Fig. 6. According to the graph, double bond of oleic acid which can be detected at the wavelength of 2980 cm^{-1} , disappeared when epoxide groups are formed. The functional groups of oxirane ring opening (C–O–C) was detected at the wavelength of 1340 cm^{-1} .²² Besides, alcohol groups (OH) and groups of polyols are present at the wavelengths of 3440 and 816 cm^{-1} , respectively. The polyols groups were obtained after degradation of oxirane ring occurred at the wavenumber of 1340 cm^{-1} .

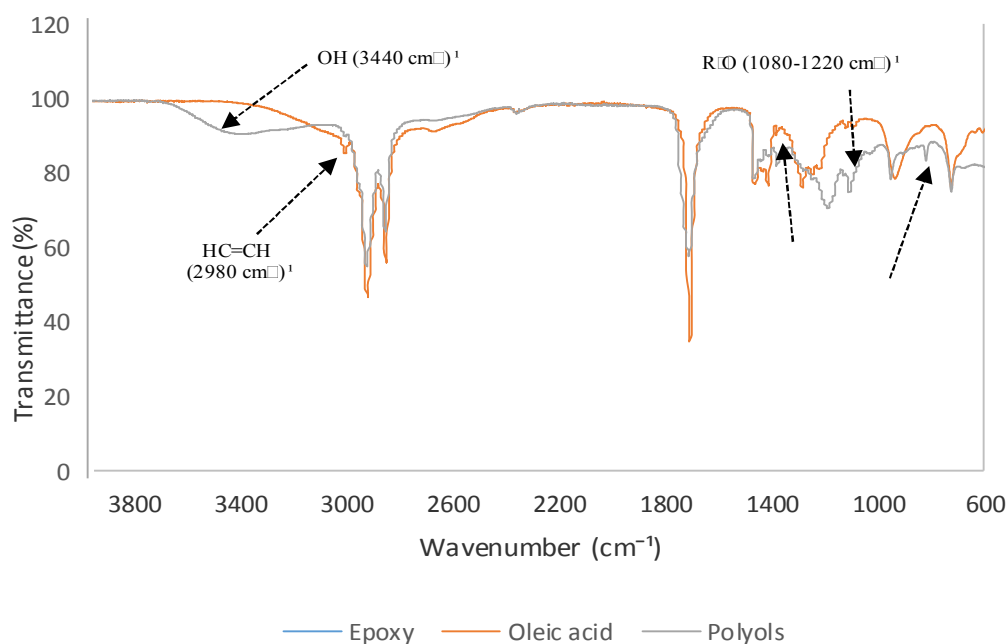


Fig. 6. FTIR combination of oleic acid, epoxide and polyols

4. Conclusions

The investigation of optimization involves three parameters: temperature, molar ratio of formic acid to oleic acid and of hydrogen peroxide to oleic acid. A set of 20 experiments was run to determine the percentage of RCO and to identify the optimum values of temperature, molar ratio of formic acid and hydrogen peroxide – 318 K, 1.64:1 and 2.0:1, respectively. Linear regression was applied to ensure success relationship between the predicted and the actual ones. The value of R^2 was 0.9958 which implies that 99.58% of actual data agreed well with the predicted data. Besides, analysis of variance (ANOVA) was used to identify the significant parameters and sources of model which involved linear, square and two-way interaction. All the parameters and models were significant because of satisfactory p -value, which is less than 0.05 for linear model and 0.0001 for the square and

two-way interactions models. The optimum reaction conditions were successfully obtained by RSM. FTIR analysis confirmed the formation of epoxy functional at the wavelength of 1340 cm^{-1} . Also, formation of polyols from epoxidized oleic acid under optimum conditions were proven by FTIR analysis at the wavelength of 816 cm^{-1} .

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ОПТИМІЗАЦІЯ ПРОЦЕСУ ЕПОКСИДУВАННЯ ПАЛЬМОВОЇ ОЛЕЙНОЇ КИСЛОТИ ДЛЯ ОДЕРЖАННЯ ПОЛІОЛІВ

Анотація. За допомогою методу крутого сходження (методу RSM) на основі трирівневого трифакторного експерименту (CCD) проведено оптимізацію процесу епоксидування. З метою визначення оптимальних умов реакції для отримання поліолів вивчено відгук відносного вмісту оксирану (%RCO). Прогнозоване значення моделі (85%) відмінно узгоджувалось з експериментальним значенням (81%). Встановлено, що всі параметри (температура, молярне співвідношення мурашиної кислоти до олеїнової кислоти та пероксиду водню до олеїнової кислоти) мали суттєвий вплив на хід реакції епоксидування ($p < 0,05$). Показано, що взаємодія між усіма параметрами має велике значення і при $p < 0,0001$. За методом RSM встановлені оптимальні умови реакції: температура 318 K, молярне співвідношення мурашиної кислоти до олеїнової кислоти 1,64:1 та молярне співвідношення пероксиду водню до олеїнової кислоти 2:1. Епоксидування пальмової олеїнової кислоти здійснювалося з використанням *in situ* пероксимурашиної кислоти. За допомогою Фур'є-спектроскопії доведено утворення епоксидних функціональних груп за оптимальних умов реакції при довжині хвилі 1340 см⁻¹. Ця епоксидна група була використана для отримання поліолів за допомогою процесу гідроксилювання, а функціональна група поліолів була виявлена при довжині хвилі 816 см⁻¹.

Ключові слова: температура, молярне співвідношення, мурашина кислота, пероксид водню, епоксидування, поліолі.