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Optimization of the Epoxidation Process Parameters of Fatty Acid Methyl Esters using Response Surface Methodology (RSM) based on Central Composite Design (CCD)

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ABSTRACT

The aim of this study was to apply the experimental design methodology in the optimization of the epoxidation of fatty acid methyl esters (FAME) derived from Jatropha oil. This experiment was carried out with peracetic acid generated in situ by using hydrogen peroxide and acetic acid. The reaction surface (RSM) methodology based on the central composite design (CCD) approach is applied, which involves the percentage (%) of the epoxidation yield as the reaction variables. The reactions were described as the function of parameters such as temperature (50-80 °C), mol ratio of hydrogen peroxide (HP) to unsaturation (1.1-2 mol), mol ratio of acetic acid (AA) to unsaturation (0.5-0.8 mol) and time (2-7 hours). The optimum percentage of epoxidation yield (90.98 %) was at the condition of 65 °C reaction temperature, HP to unsaturation mol ratio of 2.19, AA to unsaturation mol ratio of 0.65 for 6 hours. The formation of epoxides product (oxirane) was confirmed using Fourier transform infrared spectroscopy oxirane peaks (doublet) at 825 and 843 cm^{-1}*. The result showed good agreement with the predicted values from the RSM model.*

Keywords: Fatty acid methyl esters; Jatropha, epoxidation; optimization; response surface methodology

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INTRODUCTION

The search for alternative materials for the chemical industry is gaining attention due to decreasing petroleum resources and increasing environmental awareness associated with these non-renewable resources. These problems urge researchers to find an abundant and environmentally friendly alternative to promote greener chemistry. Renewable resources have been identified globally as effective alternatives to replace feedstock for the chemical industry. One of the renewable resources identified is plant oil. Due to their high availability, they are cost-effective products and can be chemically modified to improve their properties. Various research has been conducted concerning this matter, such as cottonseed oil [1], vernia oil [2], sesame oil [3], jatropha oil [4], and soybean oil [5]. Attention is directed to non-edible oil to avoid any challenge or competition with the food industry. Therefore, in this research, jatropha oil is selected as feedstock as it provides no or less benefit to human or animal consumption because it is poisonous.

The physicochemical properties of plant oils depend on the composition of fatty acid content. Their iodine value (IV) indicates that unsaturation or double bonds contribute to unique chemical structures that allow multiple chemical modifications to improve their characteristics [6,7]. Bio-polymer derived from plant oils possess exciting attributes such as being highly biodegradable, ease of processing, contributing no or less environmental pollution, having low production costs, and being non-toxic [8,9]. Owing to these properties, they are highly preferred to be utilized as an alternative to non-renewable resources [10,11,12].

The epoxidation process is one of the simplest and most widely used methods to modify plant oil structures. This approach applies the conversion method of double bonds in oils by incorporating an oxygen atom and forming a product called epoxy. Epoxides contain oxirane rings, making them highly reactive and applied in various industrial applications such as adhesives, plasticizers, bio-lubricant, and bio-based epoxy resins [10,13]. Methods of epoxidation, temperature, the mol ratio of hydrogen peroxide to unsaturation, the mol ratio of organic acid to unsaturation, reaction time, stirring speed, and the presence and type of catalyst are the factors that need to be considered while utilizing the epoxidation process [14,15]. This process is conducted with organic acid (formic or acetic acid), hydrogen peroxide, and catalysts. Organic acids react with hydrogen peroxide to produce peroxy acids, either in-situ formed or ex-situ peroxy acids, with or without the presence of catalysts [3,16]. The reaction between hydrogen peroxide and acetic acid formed peroxy acids, and it plays an essential role as the double bond oxidizing agent. As a result, compounds with more excellent oxidation stability, lower acidity, and more remarkable ability to be adsorbed by metal surfaces are synthesized [15].

There is minimal information currently available regarding the use of fatty acid methyl esters (FAME) for epoxide synthesis with response surface methodology (RSM)[17]. RSM can optimize the epoxidation of ricinoleic acid methyl ester in the presence of the phase-transfer catalyst by applying the Box-Behnken technique. Similar work was also carried out by Hernández-Cruz et al. [15] when they also performed the Box-Behnken technique to optimize the epoxidation reaction of chicken fat with peracetic acid. Another work conducted by Aguele et al. [18] also utilized a similar technique of RSM in optimizing the Huracrepitan seed oil. The methods used in this research are similar to the earlier mentioned methods. The optimization study of the epoxidation of FAME is using Central Composite Design (CCD) technique as a minor modification in the RSM method. RSM has a variety of optimization designs, but the same concept of epoxidation is applied. The findings from the previous research emphasized that hydrogen peroxide had the most significant effect on the degree of epoxidation.

This project aims to develop a framework to investigate the significant factors involved in epoxidation reaction: reaction temperature, the mol ratio of hydrogen peroxide (HP) to unsaturation, and the mol ratio of organic acid, which later will be addressed as acetic acid (AA) to unsaturation, and reaction time on the epoxidation reaction. Firstly, as a practical technique of RSM, CCD was implemented as an optimization tool to identify the optimal condition and investigate the relationship between the reaction variables. Subsequently, the formed epoxides were characterized using Fourier Transform Infra-Red (FTIR).

METHODOLOGY

Materials and Method

Jatropha oil used in this research was acquired from Bionas Development Berhad without further purification. Hydrogen peroxide (50 %) was purchased from Qrec Sdn Bhd, glacial acetic acid from R&M. These chemicals were used without further purifications. Production of FAME was prepared through transesterification, conducted in a 1000 ml tree-necked flask equipped with a mechanical stirrer, reflux condenser, and thermometer under atmospheric pressure. The epoxidation process was executed with peroxy acid generated in-situ, using acidic ion exchange resin (Amberlite IR-120) as a catalyst [19]. Figures 1 and 2 illustrate the transesterification process and the equation. Meanwhile, Figures 3 and 4 illustrate the transesterification process and the equation.

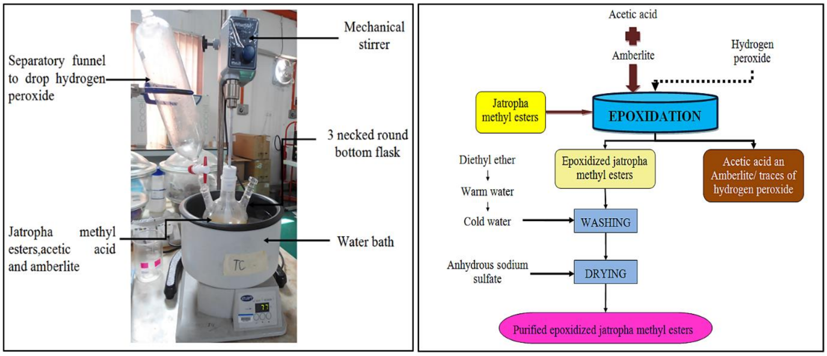

Figure 1:Transesterification process

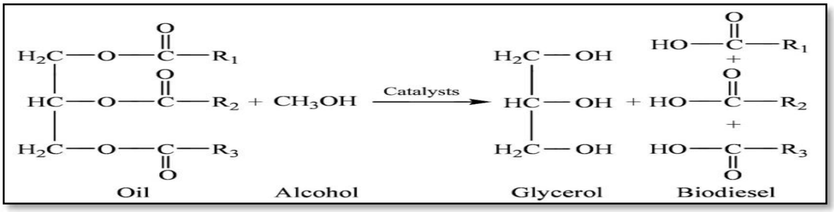

Figure 2: Transesterification reaction of a triglyceride with alcohol

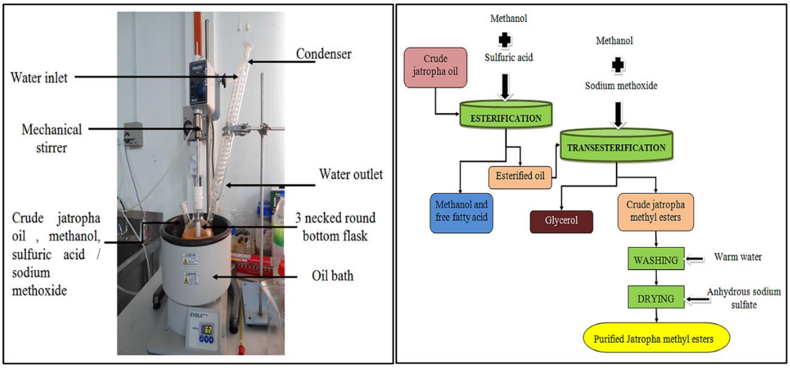

Figure 3: Epoxidation process

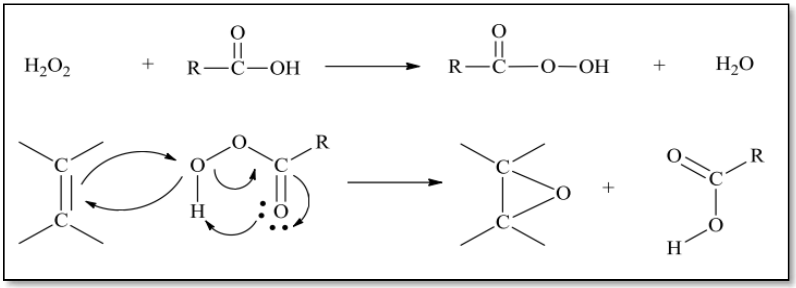

Figure 4: Epoxidation using in-situ production of the peracetic acid process

Design of experiment

Preliminary studies were conducted in reported literature varying the value of temperature, the mol ratio of hydrogen peroxide to unsaturation, mol ratio of acetic acid to unsaturation, and reaction time. The graph of each parameter with respect to epoxidation yield was plotted to identify the optimum values [19]. Methods of calculating the mol ratio were taken from Manthey [20]. Optimization studies in this research were conducted following the reported preliminary results. In this study, only the four most significant parameters were investigated: reaction temperature, the mol ratio of HP to unsaturation, and AA ratio to unsaturation and reaction time. Stirring speed and catalyst loading were maintained constant for all experiments at 1500 rpm and 16 % from the total weight of the sample, respectively. For each experiment (run 1-run 30), 50 ml samples were withdrawn at regular intervals, washed, and analyzed for oxirane oxygen content, indicating the epoxidation yield. The washing process was conducted using 5 % diethyl ether and warm and cold water. Firstly, the bio-epoxides were washed with successive portions of diethyl ether, followed by cold water and finally hot water, to remove residual fatty acid or unreacted chemicals. The solution was dried over anhydrous sodium sulfate (1.5 % weight of sample: weight of diethyl ether) overnight in an oven at 70 °C to remove any traces of water.

Four reaction parameters were studied in thirty experiments based on response surface methodology (RSM) in central composite design (CCD). The values of studied parameters are presented in Table 1.

Table 1 : Reaction variables levels

Parameters	Symbol	Unit	Variable levels	
			-1 level	+1 level
Temperature	A	°C	50	80
Mol ratio HP	B	Mol	1.1	2
Mol ratio AA	C	Mol	0.5	0.8
Time	D	Hr	2	7

Analytical Analysis

Oxirane Oxygen Content

The response of this study is the epoxidation yield indicated by the value of the oxirane oxygen content (OOC). Determining its value was crucial in determining the quality of epoxides and the efficiency of the epoxidation process. The OOC of each sample was determined using the direct method with a hydrobromic acid solution. Methods of calculation of selected data were taken from Paquot [21].

FTIR Spectroscopy Analysis

The Fourier Transform Infra-Red (FTIR) analysis method was adopted from the previous study [22,23]. This analysis aims to determine the functional group in the Jatropha oil, FAME, and epoxides. A potassium bromide (KBr) pallet was used to determine the background signal. The spectra were obtained over the frequency range 4000-650 cm^{-1} at the resolution of 4 cm^{-1}, and the final output was in % transmittance. The spectra were recorded on an FTIR Perkin-Elmer spectrophotometer model Spectrum-1000 (Perkin-Elmer, Norwalk, CT, USA).

RESULTS AND DISCUSSION

ANOVA Analysis

The results of all 30 runs were presented in Table 2, and analysis was conducted using ANOVA techniques. Experimental values and predicted values are shown as well. The response of the experiment is the epoxidation yield, which is calculated based on the percentage conversion of unsaturation to oxirane ring. The highest epoxidation yield was 90.98 % obtained at reaction temperature 65 °C, the mol ratio of hydrogen peroxide 2.19 mol, acetic acid 0.65 mol within 4.5 hours reaction time (Run 14). On the other hand, the lowest epoxidation yield was 23.70 %, obtained at a reaction temperature of 50 °C, the mol ratio of hydrogen peroxide 1.1 mol, acetic acid 0.5 in 2 hours (Run 3).

Table 2: Experimental design matrix and results of epoxidation yield of FAME as affected by temperature, the mol ratio of HP, AA, and time

Run No	Temp (A, °C)	Mol ratio HP (B, mol)	Mol ratio AA (C, mol)	Time (D, hr)	Epoxidation yield (%)	
					Experimental value	Predicted value
1	50	2.00	0.50	2.0	38.35	38.52
2	50	1.10	0.80	7.0	62.77	63.13
3	50	1.10	0.50	2.0	23.70	23.39
4	65	1.55	0.65	4.5	89.26	88.76
5	80	2.00	0.50	2.0	65.72	64.87
6	65	1.55	0.65	4.5	88.67	88.76
7	65	1.55	0.44	4.5	68.83	69.63
8	80	1.10	0.50	2.0	43.89	43.64
9	80	2.00	0.50	7.0	65.50	65.40
10	50	1.10	0.50	7.0	77.54	77.62

11	65	1.55	0.65	8.0	84.44	83.44
12	80	2.00	0.80	2.0	89.73	90.18
13	50	2.00	0.50	7.0	64.20	64.48
14	65	2.19	0.65	4.5	90.98	90.86
15	86	1.55	0.65	4.5	68.97	70.03
16	65	1.55	0.65	4.5	88.79	88.76
17	65	1.55	0.86	4.5	78.16	77.28
18	65	1.55	0.65	4.5	87.73	88.76
19	65	1.55	0.65	1.0	65.29	66.21
20	50	2.00	0.80	2.0	64.42	64.06
21	65	1.55	0.65	4.5	88.26	88.76
22	80	1.10	0.50	7.0	72.58	72.45
23	44	1.55	0.65	4.5	56.37	55.23
24	80	1.10	0.80	7.0	57.37	57.72
25	80	1.10	0.80	2.0	60.09	59.31
26	50	1.10	0.80	2.0	38.66	39.29
27	50	2.00	0.80	7.0	58.84	59.62
28	65	0.91	0.65	4.5	78.28	78.33
29	65	1.55	0.65	4.5	89.67	88.76
30	80	2.00	0.80	7.0	60.48	60.30

Analysis of variance (ANOVA) is shown in Table 3, where A represents the temperature, B represents the mol ratio of HP, C represents the mol ratio of AA, and D represents the reaction time. The P-value for all four parameters is <0.0001 indicated that all reaction parameters were significant in the epoxidation reaction. The effect of a combination of two parameters on epoxidation yield was significant except the combination of temperature and mol ratio of acetic acid as depicted by the p-value >0.0001, which is 0.7929. The significance of individual experimental parameters can be judged based on the corresponding p-values. The lower the p-values, the higher the importance of the corresponding coefficient will be. Besides, p-values also indicate the strength of interaction between cross-products of variables. The effect of these parameters on epoxidation yield can be observed by considering the F-value and p-values shown for each parameter in Table 3. The higher the F-value (and the lower p-values), the more significant the effect of the parameters on epoxidation yield [24,25].

Table 3: Analysis of variance (ANOVA) for the fitted quadratic polynomial model

Sources of variations	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	8946.066	14	639.004685	844.6024	< 0.0001
A-Temperature	547.9903	1	547.990287	724.3044	< 0.0001
B-Mol ratio HP	392.5025	1	392.502538	518.789	< 0.0001
C-Mol ratio AA	146.2032	1	146.203186	193.2436	< 0.0001
D-Time	741.8416	1	741.841572	980.5267	< 0.0001
AB	37.11856	1	37.118556	49.06133	< 0.0001
AC	0.054056	1	0.05405625	0.071449	0.7929
AD	646.6849	1	646.6849	854.7537	< 0.0001
BC	92.83323	1	92.833225	122.702	< 0.0001
BD	799.617	1	799.617006	1056.891	< 0.0001
CD	924.312	1	924.312006	1221.706	< 0.0001
A^2	1592.293	1	1592.29263	2104.608	< 0.0001
B^2	40.43799	1	40.4379943	53.44879	< 0.0001
C^2	546.0672	1	546.067209	721.7626	< 0.0001
D^2	452.7714	1	452.771433	598.4492	< 0.0001
Residual	11.34862	15	0.75657455		
Lack of Fit	8.956018	10	0.89560183	1.871608	0.2535
Pure Error	2.3926	5	0.47852		
Cor Total	8957.414	29			
	CV %= 0.87	R^2= 0.9987	R^2 adj= 0.9976	R^2 predicted =0.9943	

For a model to be considered fit for its reliable application, a high statistical significance is required. The high significance of a model was indicated by a high F-value, 844.6024, at a very low probability value. The calculated probability value (p-value) from the analysis of the model is <0.001 ($P<0.05$ indicated that model terms were significant). Therefore, the Model F-value of 844.6024 implied the model was significant. There was only a 0.01 % chance that a "Model F-value" this large could occur due to noise. In addition, the lack of fit condition for the model is not significant indicated by the P-value>0.05. This result agrees with a discussion by Zhao et al. [17] in their reported research.

The model's fitness was evaluated by calculating the regression equation and the multiple correlation coefficient R^2. A high value of R^2 =0.9987 indicated that 99.87 % of the response variables (epoxidation yield) were dependent on the independent variables (A, B, C, and D), and only 0.13 % of total variations were dependent on other variables, not defined by the models. Therefore, this model could be used with high reliability to predict results with good precision indicated by a high R^2 value [26,27]. Furthermore, low coefficients of variance, CV=0.87, indicated high precision, low scatter, and better repeatability in experimental results. In addition, a high value of the adjusted determination coefficient (Adj R^2=0.9976) also showed the high significance of the model.

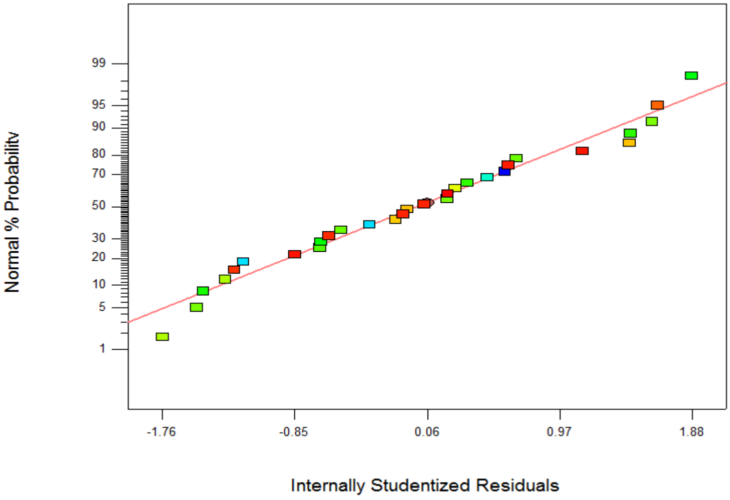

Figure 5: Normal probability plot of internally studentized residuals

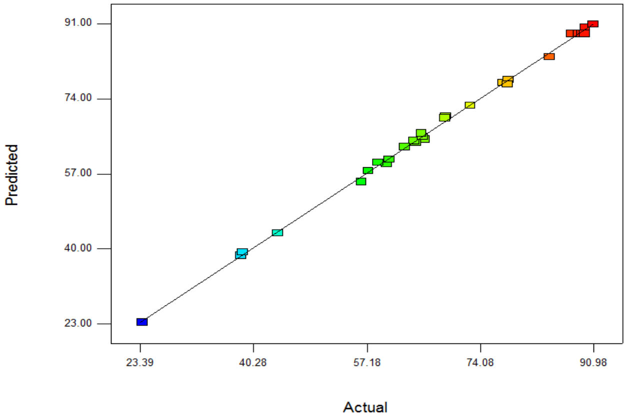

Figure 6: Comparison of actual and predicted values of the response

The normal plot of residuals is shown in Figure 5 and indicates normal distributions of the residuals. The points should follow a straight line with an acceptable scatter if their distribution is normal. Based on the plot, it was demonstrated that the data distribution was substantially normal. In Figure 6, a plot of actual values against the values predicted by the model is presented. Most of the data points were distributed evenly along the line showing a high value for R^2 (0.9987), and experimental results and the predicted values were in excellent agreement [17].

Influence of process variables on maximum epoxidation yield

Three-dimensional response surface plots were obtained from design expert software, and the graphical presentation is shown and discussed below. Two reaction parameters were varied to get this plot while keeping the other two parameters at a central level. Figures 7-12 displayed the three-dimensional response surface plot describing the cross effect of time and temperature on the maximum epoxidation yield concerning other process variables.

Effect of temperature and mol ratio of hydrogen peroxide on epoxidation yield

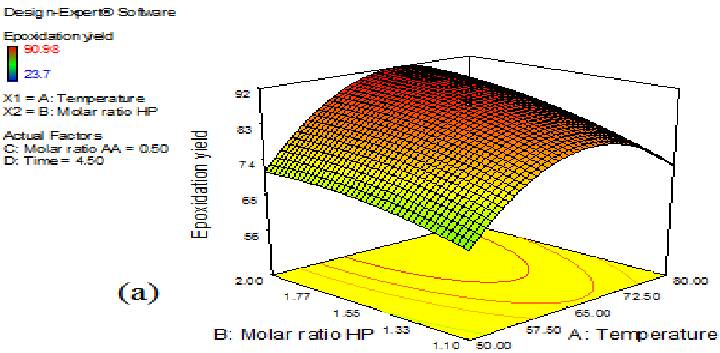

Figure 7: Response surface plots for the effect of the temperature (A) and the mol ratio of hydrogen peroxide (B) on the maximum epoxidation yield

Figure 7 illustrates the effect of temperature (A) and the mol ratio of hydrogen peroxide (B) on the epoxidation yield using a 3D response surface. During the epoxidation process, to study their combined interactions, the mol ratio of acetic acid and reaction time were kept constant at a value of 0.5 moles and 4.5 hours, respectively. The epoxidation yield increased with an increase of reaction temperature from 50 °C to 80 °C. These results agree well with existing studies on epoxidation of ricinoleic acid methyl ester [16], which stated that the reaction rate increased at higher temperatures due to increased collision rates between the reactants. The increasing trend was observed up to 66 °C. Beyond this temperature, depletion of epoxidation yield was noticed. As shown in the figure, one can see that an increase in the value of epoxidation yield increased when the mol ratio of hydrogen peroxide was elevated from 1.1 to 2 moles. However, deterioration of epoxidation yield was noticed at 1.55 moles of hydrogen peroxide at 65 °C. Previous research mentioned that the rate of epoxidation increased progressively with increased hydrogen peroxide concentrations; however, the stability of the oxirane ring is reduced [28,29].

On the other hand, Liew et al. [30] stated that at lower concentrations of hydrogen peroxide, oxirane ring and oxirane ring cleavage formations coincide. Hence, a moderate concentration of hydrogen peroxide was suitable to achieve the maximum epoxidation yield. Therefore, temperature 65 °C and 1.55 moles of hydrogen peroxide were considered optimum conditions for further experimentations.

Effect of temperature and mol ratio of acetic acid on epoxidation yield

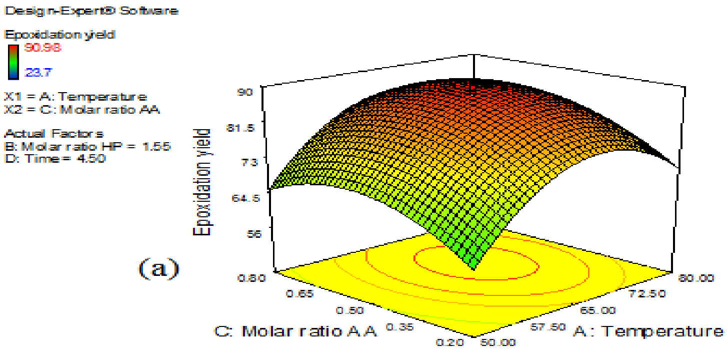

Figure 8: Response surface plots for the effect of the temperature (A) and the mol ratio of acetic acid (C) on the maximum epoxidation yield

A representation of a 3D response surface plot describing the effect of temperature (A) and the mol ratio of acetic acid (C) on the maximum epoxidation yield is illustrated in Figure 8. The effect of the mol ratio of acetic acid was studied in the range between 0.2 to 0.8 moles. Maximum epoxidation yield was achieved at 0.5 moles, and further increased value resulted in the depletion of epoxidation yield. Based on the demonstrated trends of this figure, it can be concluded that epoxidation yield increased linearly with an increase in both temperature and mol ratio of acetic acid. However, when both values reached 60 °C and 0.5 moles, respectively, epoxidation yield experienced a reduction due to oxirane cleavage. The result has a similar performance with a previous report [29] studying the epoxidation of fatty acid methyl esters from waste cooking oil. Therefore, the moderate temperature at 65 °C was considered suitable for epoxidation of FAME.

Effect of temperature and time on epoxidation yield

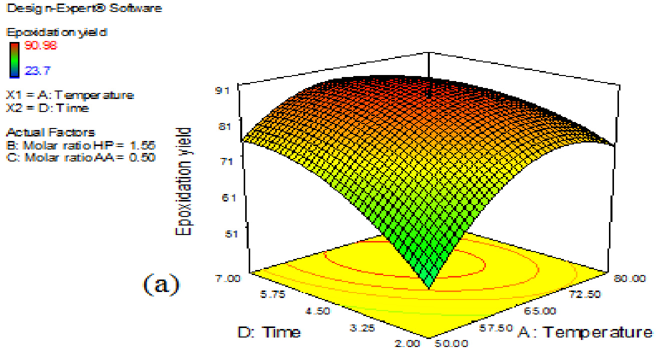

Figure 9: Response surface plots for the effect of the temperature (A) and time (D) on the maximum epoxidation yield

The effect of varying temperature (A) and time (D) on the maximum epoxidation yield concerning other reaction parameters is demonstrated in Figure 9. As illustrated, epoxidation yield increased as the reaction time increased from 2 to 8 hours. This finding is consistent with the previous findings [31], suggesting that epoxidation yield increases gradually with time. However, the increasing trend was observed up to a specific time, which was 4.5 hours. Higher reaction time provided an opportunity for oxirane rings to react with excess hydrogen peroxide, acetic acid, and water-produced glycol [3,32]. As shown in Figure 9, the epoxidation yield achieved maximum value at 65 °C at a reaction time of 4.5 hours. Beyond this value, depletion of epoxidation yield was noticed. Thus, prolonged reaction time and increased reaction temperature reduced epoxidation yield due to oxirane cleavage. Similar results were discussed by Mushtaq et al. [24] during the epoxidation of FAME. Their study reported a shorter reaction time due to the usage of formic acid as an oxygen carrier and a higher concentration of hydrogen peroxide.

Effect of mol ratio of hydrogen peroxide and the mol ratio of acetic acid on epoxidation yield

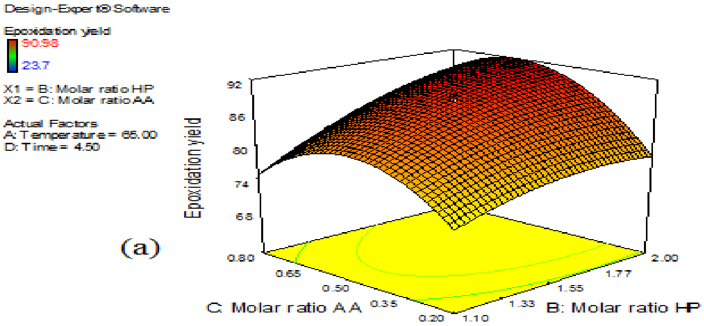

Figure 10: Response surface plots for the molar ratio hydrogen peroxide (B) and molar ratio acetic acid (C) on the maximum epoxidation yield

Figure 10 shows the comparison of the mol ratio of hydrogen peroxide (B) and acetic acid (C) on the epoxidation yield, represented using the 3D response surface plot. The mol ratio of acetic acid is investigated between 0.2 to 0.8 moles. The increasing the value of mol ratio increased the epoxidation yield, and maximum yield was obtained at unsaturation: mol ratio acetic acid: mol ratio hydrogen peroxide 1:0.5:1.5 moles, respectively. Acetic acid plays both functions in forming the oxirane ring and reactant in the hydrolysis of the oxirane ring [15]. Acetic acid and hydrogen peroxide play a vital role in generating peroxyacetic acid to form the oxirane ring. The presence of a 15 % (wt %) catalyst accelerates the formation of peroxyacetic acid. During the reaction, the acetic acid concentration is maintained, regenerated in the epoxidation process, and plays a less significant role than hydrogen peroxide [33].

Effect of mol ratio of hydrogen peroxide and time on epoxidation yields

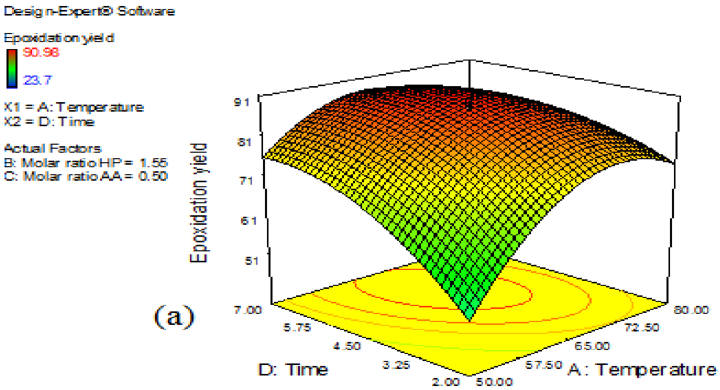

Figure 11: Response surface plots for the molar ratio hydrogen peroxide (B) and time (D) on the maximum epoxidation yield

The relationship between the mol ratio of hydrogen peroxide (B) and time is presented in Figure 11. The mol ratio of hydrogen peroxide was varied from 1.1 to 2.0 moles. The result showed that epoxidation yield also increased due to the formation of more peroxyacetic acid. [3]. The highest epoxidation yield was obtained at a mol ratio of 1.55. However, at higher concentrations and longer time, epoxidation yield was similar. Therefore, due to the high possibility of an explosion at higher concentrations of hydrogen peroxide, it is not recommended [23]. In addition, a paper reported by previous researchers [32] stated that the shortcomings of using high concentrations of hydrogen peroxide also included the problem in agitation and decreased the mass transfer rate, which resulted in low epoxidation yield. Based on figure above, it can be concluded that the optimum mol ratio of hydrogen peroxide and reaction time was 1.55 moles and 4.5 hours.

Effect of mol ratio of acetic acid and time on epoxidation yield

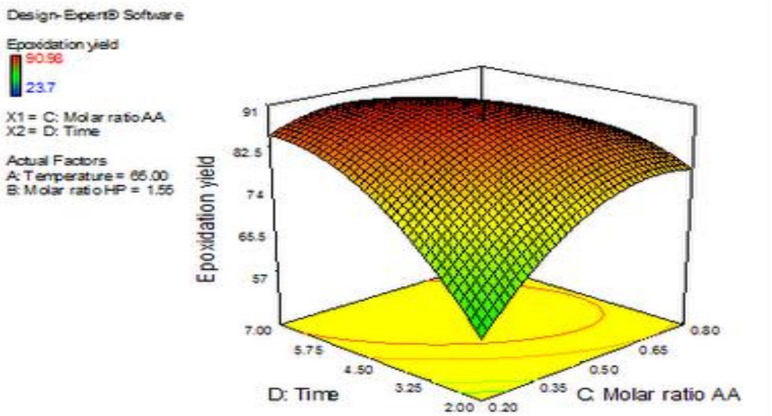

Figure 12: Response surface plots for the effect of the mol ratio acetic acid (C) and time (D) on the maximum epoxidation yield

The results of the 3D surface plot of the relationship between acetic acid (C) and time (D) on epoxidation yield are provided in Figure 12. The effect of acetic acid was less significant than reaction time. Although increasing acetic acid concentrations increased epoxidation yield, the oxirane cleavage was less apparent due to low concentrations of acetic acid used [28]. However, reaction time was more significant in the epoxidation reaction, as shown in the figure. At the start of reaction time, epoxidation yield was lower, increasing as the reaction time increased. Maximum epoxidation yield was obtained at a reaction time of 4.5 hours. Beyond this point, the depletion of the oxirane ring was observed.

Characterization using FTIR

The spectroscopic properties of Jatropha oil, FAME, and epoxides were studied by FTIR spectroscopy. FTIR is a sensitive technique for monitoring FAME and oxirane ring formation in transesterification and epoxidation. FTIR spectra demonstrated a peak between 3002-3008 cm^{-1} associated with the stretching of C=C bonds. As shown in Figure 13, the presence of unsaturation in Jatropha oil and FAME was confirmed by the peak at 3008 cm^{-1}. Similar findings were reported by Mai et al. [34] in the study of the production of bio-epoxy resin from Jatropha oil.

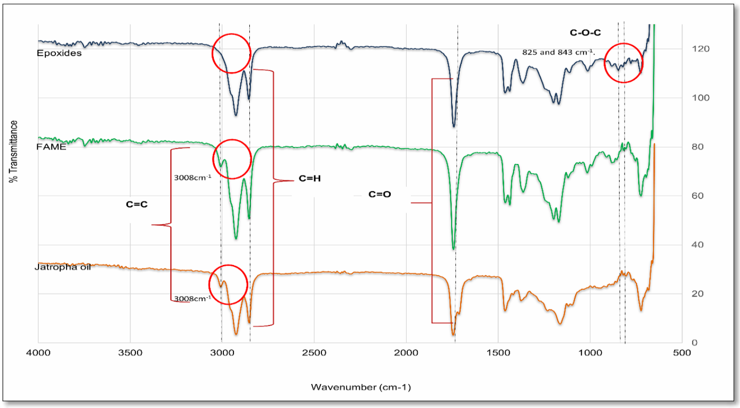

Figure 13: FTIR spectra of Jatropha oil, FAME, and epoxides

After completing the transesterification process, the unsaturation peak was still present. However, the IR spectrum of Jatropha oil showed the ester linkage of triglyceride at 1744 cm^{-1}, whereas the peak shifted to 1743 cm^{-1} in FAME. This change reduction of the carbonyl group from three to only one in FAME. Similar results were reported by [35,36]. The formation of the oxirane ring after the epoxidation process was also demonstrated in Figure 13. It can be observed that the disappearance of the peak at 3008 cm^{-1} in FAME and the appearance of oxirane peaks (doublet) at 825 and 843 cm^{-1}. This result was following reports of previous works that oxirane ring exist in the range of 820 to 843 cm^{-1} [2,8,37]. Complete disappearance of the peaks indicated a high conversion of unsaturation to oxirane ring, which is 90.98 %. The absence of hydroxyl peaks in the range of 3000-3500 cm^{-1} (O-H range) proved that minimum oxirane cleavage occurred in the epoxidation process during the optimum conditions. These results agreed with an investigation conducted by Liew et al. [30] that hydroxyl and unsaturation peaks must not appear in the FTIR spectrum to optimize epoxidation yield.

CONCLUSION

The epoxidation reaction of FAME with peroxy acids was successfully optimized, with the stirring rate maintained at 1500 rpm and catalyst loading of 16 %. RSM was found to be a helpful technique for optimizing the epoxidation of FAME. The optimal condition achieved with the percentage of epoxidation yield (90.98 %) was at 65 °C reaction temperature, HP to unsaturation mol ratio of 1.55, AA to unsaturation mol ratio of 0.5 for 4.5 hours. Under these conditions, the experimental yield was 90.98 % against the predicted yield of 90.86 %. Hence, with minimal error, the statistical analysis results showed that the process parameters (temperature, mol ratio of HP to unsaturation, mol ratio of AA to unsaturation, and reaction time) significantly affect the response.

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