

## Epoxidation of melon seed (*Citrullus colocynthis L*) oil: A kinetic and thermodynamic approach for industrial applications

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### ABSTRACT

Unsaturated melon seed oil had its epoxidation process studied at varied temperatures (30°C -70°C) within a three hours reaction time, for possible oxirane formation because of its industrial significance. Oil was extracted from melon seeds and characterized for its physicochemical properties using standard methods. The specific gravity (30°C), iodine value, acid value, saponification value, peroxide value and percentage oil content were found as 0.931, 118.69 gI<sub>2</sub>/100g, 5.08 mg KOH/g, 165.7 mg KOH/g, 4.75 meq/kg and 60% respectively. Physicochemical characteristic of the oil reveals it is semi-drying oil which is rich in high molecular weight fatty acids, with good stability against rancidity. The epoxidation process which was monitored by its oxirane content is seen to proceed faster at the highest studied temperature of 70°C, giving the highest oxirane content of 5.87%. Optimization of the epoxidation process showed that optimum epoxidation of melon seed oil can be achieved at 70°C within one hour; as compared to other temperatures studied. Process kinetics evaluated in terms of its oxirane yield showed that the epoxidation process followed pseudo-zero order kinetics with respect to oxirane formation. Their rate constants were found to be 0.032 hr<sup>-1</sup>, 0.041 hr<sup>-1</sup>, 0.059 hr<sup>-1</sup> and 0.24 hr<sup>-1</sup> for the epoxidation process at 30°C, 50°C, 60°C and 70°C respectively; which shows that the epoxidation rate increases with increasing temperature. Thermodynamic parameters such as activation energy (E<sub>a</sub>), activation enthalpy (ΔH<sup>‡</sup>), activation entropy (ΔS<sup>‡</sup>) and Gibb's free energy (ΔG<sup>‡</sup>) were determined to be 133.11 kJmol<sup>-1</sup>, 18.22 kJmol<sup>-1</sup>, -0.21 kJmol<sup>-1</sup> K<sup>-1</sup> and 87.95 kJmol<sup>-1</sup> respectively. The observed thermodynamic properties indicated strong temperature dependence of the epoxidation reaction rate, endothermicity and non-spontaneity of the process. The findings of this study will guide in maximizing oxirane yield and save cost significantly in the production of epoxidized melon seed oil employed for large scale industrial applications.

**Keywords:** Melon seed oil, Epoxidation, Pseudo-zero order, Kinetics, Thermodynamics, Optimization.

### Introduction

Melon seeds with the scientific name *Citrullus colocynthis L*, commonly known as "Egusi" in Nigeria belongs to *Citrullus* of Cucurbitaceae

specie, consisting of a large variety generally known as melons (Mabaleha *et al.*, 2007). It is among the 300 plant species found in Africa that is quite rich in oil (53%) and protein (28%) (Ntui *et*

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al., 2009). The high protein and oil content is characteristics of Cucurbitaceae. Cucurbitaceae seeds are rich sources of oils and protein with as much as 50% oil and 35 % protein (Achu *et al.*, 2005). Major forms of epoxidizable oils are gotten from plant seeds oils. The main constituents of plant oils are triglycerides which are the product of esterification of glycerol with three fatty acids. There are several reactive functional groups and sites present in triglycerides thus providing routes for modifications, and possibilities of new structures for diverse applications. Unsaturated fatty acid found in vegetable oils can be converted into epoxy fatty acid by processes such as conventional epoxidation, catalytic acidic ion exchange resin, metal catalyst epoxidation or through the application of chemo-enzymatic epoxidation. Most recently, epoxidized vegetable oils are of high scientific interest due to the renewable nature of their precursors which are sustainable, renewable and environmentally friendly for industrial applications. Epoxidized vegetable oil serves as precursors for the synthesis of various chemicals such as carbonyl compound, polyols, glycol, lubricants, stabilizers for polymer, and plasticizers among others because of their oxirane content whose rings are of high reactivity. The unsaturated groups found in vegetable oils are modified chemically by the process of epoxidation to give value added products, thereby increasing the economic value of vegetable oils.

Researchers have studied vegetable oils as alternative feedstock to petroleum (Petrovic *et al.*, 2002; Mungroo *et al.*, 2008); hence the need for its exploration for diverse applications in the chemical industry. The study is therefore aimed at optimizing processes for the epoxidation of melon seed oil for possible industrial applications.

## Materials and Methods

### Collection of Plant Materials

Melon seeds were obtained from the local market in Okitipupa local government area of Ondo State,

South West Nigeria. Analytical grade formic acid, hydrogen bromide, hydrogen peroxide, glacial acetic acid and other chemicals were standardized and used without further purification.

### Seeds Pretreatment

The seeds were sundried for easy dehulling, after which it was dehulled manually.

### Extraction of the oil

The dried melon seeds were decorticated manually and the seed separated from the shell. The dried melon seed was milled to increase its surface area and allow for better solvent access during extraction. It was subjected to solvent extraction with n-hexane using the Soxhlet apparatus at about 60°C on a heating mantle, after which the solvent was separated from the melon seed oil by distillation. The oil obtained was weighed and the percentage oil yield was calculated using the expression below:

$$\% \text{ oil yield} = \frac{\text{weight of oil extracted}}{\text{weight of melon seed used}} * 100$$

The oil was thereafter stored in an airtight container (glass) under cool condition before use.

### Physico-chemical characterization of the oil

Acid, peroxide, iodine and saponification values were determined using standard procedures (S.M.A.O. 1994; A.O.A.C. 1995; Kardash and Tur'yan, 2005). The colour of the oil was observed by visual inspection at room temperature.

### Epoxidation process

Epoxidation process was achieved through the in situ generation of peracid, by reacting various amounts of acetic acid and hydrogen peroxide with the oil at different temperatures, using modified reports of Rangarajan *et al.* (1995) and Okiemen *et al.* (2006). Oxirane content was determined titrimetrically following standard method (Jeffery *et al.*, 1994; Aigbodion *et al.*, 1999).

### Kinetics Studies

Kinetic studies of the oil (0.13moles) epoxidation using in situ generated peracid from formic acid (0.09moles) and hydrogen peroxide (30%; 0.44moles) were carried out at 30, 50, 60 and 70°C respectively. Aliquots of the reaction mixture were collected at 30 minutes time interval as a measure for monitoring the progress of the reaction. These were placed in a separating funnel where the aqueous layer was drawn off and oil layer washed with successive portions of hexane until acid-free. The aliquot was analyzed for percentage oxirane content using already described standard methods (Jeffery *et al.*, 1994; Aigbodion *et al.*, 1999). Kinetics of epoxidation was followed as oxirane formation rate dependence on temperature (30<sup>o</sup> C -70<sup>o</sup> C)

### Results

Table 1 shows the physico-chemical characterization of the seed oil extract from melon. The oil colour been yellow is characteristic of most edible vegetable oils. Specific gravity which was found as 0.931 is in the same range (0.85–0.93) with most seed oils (Crockett, 2015; Folarin *et al.*, 2017; Akpoveta and Adetunji, 2022), indicating its less density to water; thus enabling easy flow and spreadability for industrial applications. An oil content of 60 % is seen from the seed as presented in Table 1, which agrees with earlier reports (Crockett, 2015; Akpoveta and Adetunji, 2022).

The peroxide value of 4.75meq/kg agrees with that reported for ASO (Akpoveta and Adetunji, 2022), and in line with recommended standard (Folarin *et al.*, 2017). The effect of low peroxide value is that it has the ability to resist deterioration occasioned by oxidative stress. A high peroxide value permits instability and rancidity (Folarin *et al.*, 2017). The low peroxide value of 4.75meg/kg and acid value of 5.08 mgKOH/g as seen in Table 1, indicates freshness, stability against oxidation and rancidity (Folarin *et al.*, 2017).

The acid value of 5.08 mg KOH/g is higher when compared with that reported for *Soya oil* which is 0.60mgKOH/g as well as those reported value for *C. metuliferus* and ASO (Sadou, 2007; Akpoveta and Adetunji, 2022). This is however lower than that obtained for *C. prophetarum* seed oil (Sadou, 2007). The acid value represents free fatty acid content due to enzymatic activity and this implies that the oil is not rancid.

The acid values of oil can be influenced by the nature of oil, method of extraction, maturity of seeds, storage time and storage conditions as well as moisture and high temperature which can increase the acid value due to hydrolysis of glycerides into free fatty acids (Egbekun and Ehieze, 1997).

The saponification value of melon seed oil was found as 165.70mgKOH/g as expressed in Table 1. The value is found to be lower than those for palm oil and ASO (196 – 205 mgKOH/g), olive oil (185 - 196 mgKOH/g), soyabean oil (193 mgKOH/g) and linseed oil (193 - 195 mgKOH/g) (Folkard and Sutherland, 1996; Essien and Eduok, 2013; Akpoveta and Adetunji, 2022). This indicates the richness of the oil in high molecular weight fatty acids. The iodine value of the seed oil extract from melon was found as 118.69 gI<sub>2</sub>/100g as seen in Table 1, which lies within the range of 103-128 for corn oil, 99-119 for cottonseed oil, 92-125 for mustard oil and 104-120 for sesame oil (Parthasarathy, 2014). The iodine value of the oil indicates that the oil is semi-drying oil.

Epoxidation of the oil which was observed by monitoring its oxirane content is seen to have proceeded faster with increasing temperatures as evident in the results (Table 2 and Fig. 1). It is seen from the optimization plots of oxirane yield content against time at different temperatures that the epoxidation is optimal at higher temperatures (60°C and 70°C), with highest oxirane content (5.87%) obtained at 70°C as seen in Table 2. This shows that optimum epoxidation of melon seed oil can be achieved at this temperature at a shorter time

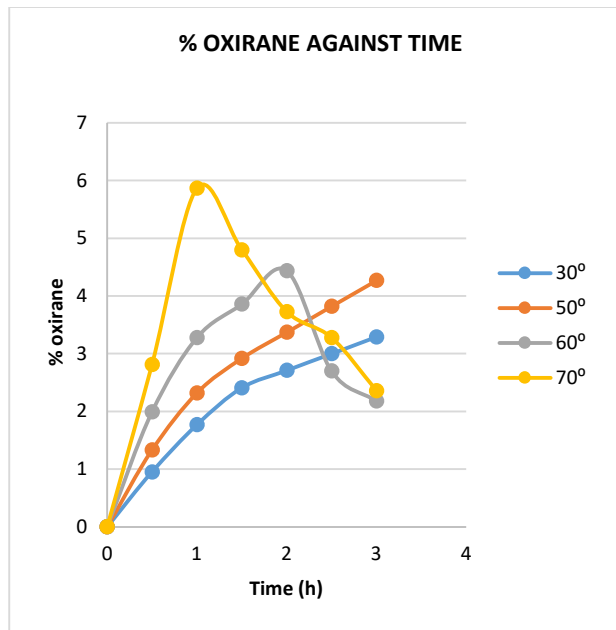
of about one hour; as compared to the other temperatures studied. A progressive decrease in oxirane content at reaction temperature of 70°C is observed at increased reaction time, indicating complete epoxidation and subsequent possible hydroxylation (ring opening reaction) of the epoxide being formed. The deviation of increased oxirane content from linearity at higher temperatures is also seen in the reports of Okiemen and Okiemen (2001) for the epoxidation of Jatropher seed oil, as well as the epoxidation of unsaturated fatty acid methyl esters (Shuangfei and Wang, 2011).

**Table 1:** Physicochemical properties of seed oil extract from melon

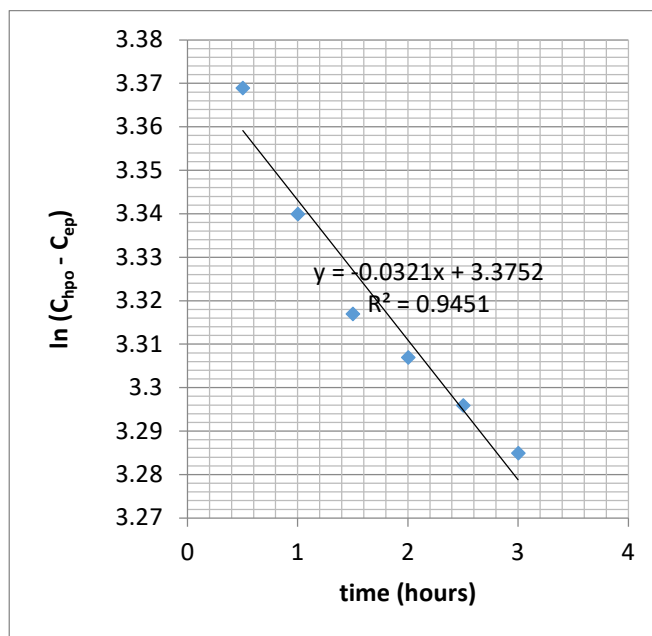
Property	Value
Colour	Yellow
Specific Gravity (30°C)	0.931
Saponification value (mgKOH/g)	165.70
Acid value (mgKOH/g)	5.08
Iodine value (gI <sub>2</sub> /100g)	118.69
% Oil Content	60
Peroxide value (meq/kg)	4.75

**Table 2:** Percentage oxirane formation at different temperatures

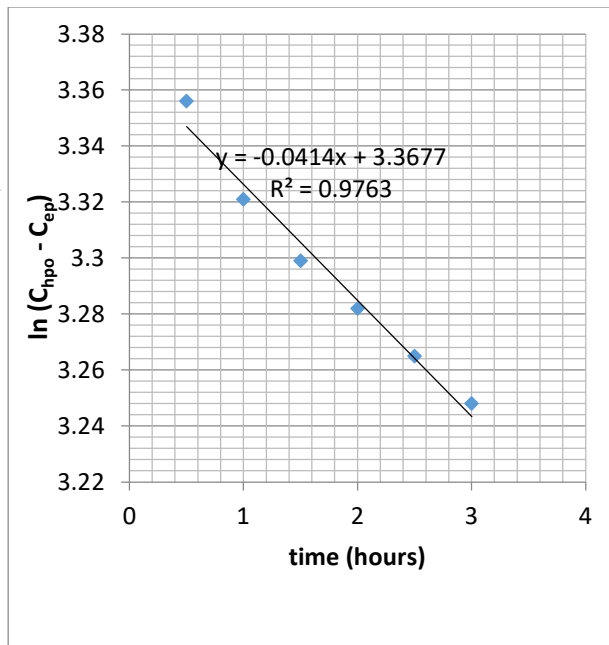
Time (hours)	% oxirane formed at 30°C	% oxirane formed at 50°C	%oxirane formed at 60°C	% oxirane formed at 70°C
0	0.00	0.00	0.00	0.00
0.5	0.95	1.33	1.99	2.81
1.0	1.77	2.32	3.28	5.87
1.5	2.41	2.92	3.86	4.80
2.0	2.71	3.37	4.44	3.73
2.5	3.00	3.82	2.70	3.28
3.0	3.29	4.27	2.18	2.36



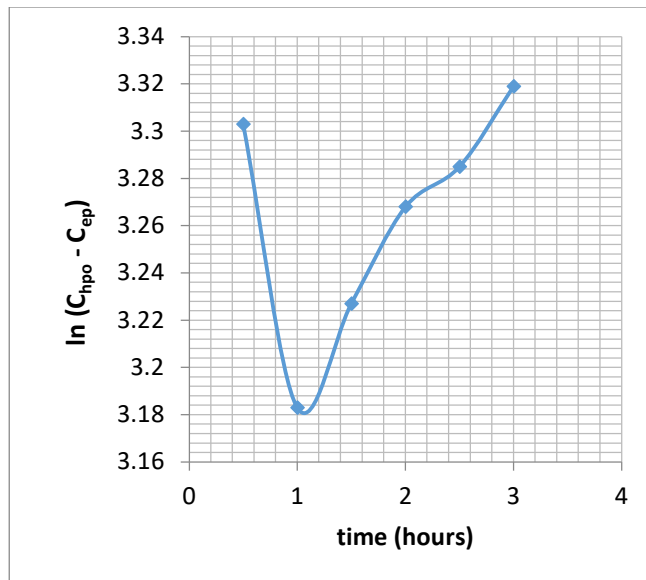
**Fig. 1:** Optimization profile for oxirane yield against time at different temperatures



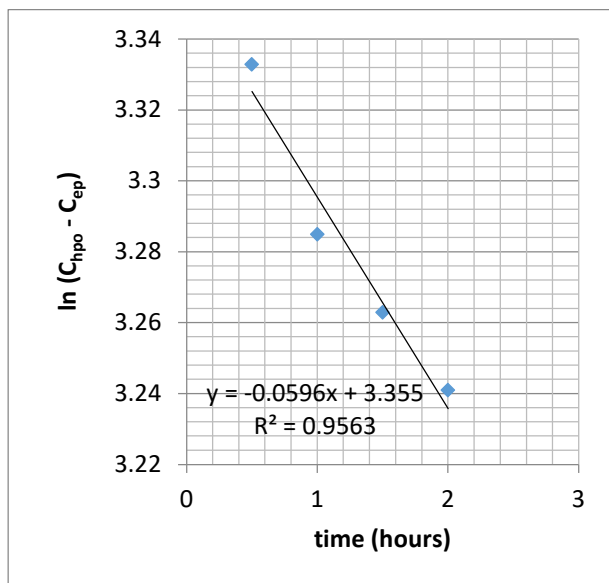
**Fig. 2:** Pseudo zero order profile for the epoxidation of seed oil extract from melon at 30°C



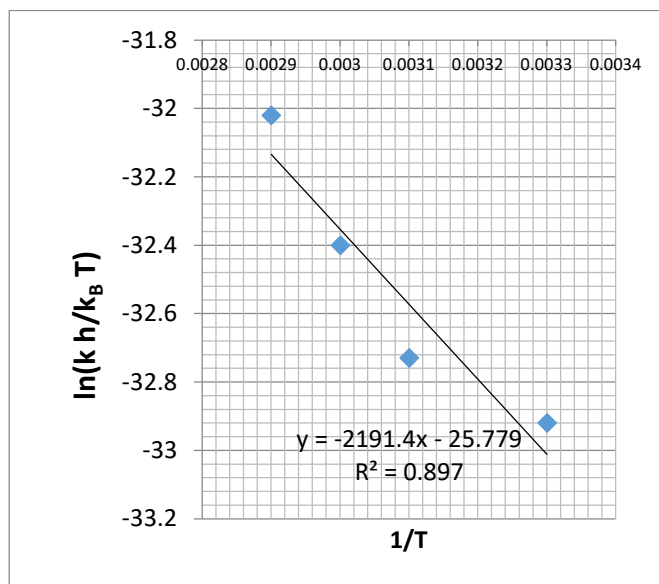
**Fig. 3:** Pseudo zero order profile for the epoxidation of seed oil extract from melon at 50°C



**Fig. 5:** Pseudo zero order profile for the epoxidation of seed oil extract from melon at 70°C



**Fig. 4:** Pseudo zero order profile for the epoxidation of seed oil extract from melon at 60°C



**Fig. 6:** Eyring plot of  $\ln(k h/k_B T)$  versus  $1/T$  for kinetics analysis of the epoxidation of seed oil extract from melon

**Table 3:** Kinetic data for the epoxidation process as a function of time at varying temperatures

Time (hours)	$\ln(C_{\text{hpo}}-C_{\text{ep}})$ (30°C)	$\ln(C_{\text{hpo}}-C_{\text{ep}})$ (50°C)	$\ln(C_{\text{hpo}}-C_{\text{ep}})$ (60°C)	$\ln(C_{\text{hpo}}-C_{\text{ep}})$ (70°C)
0.5	3.369	3.356	3.333	3.303
1	3.340	3.321	3.285	3.183
1.5	3.317	3.299	3.263	3.227
2	3.307	3.282	3.241	3.268
2.5	3.296	3.265	3.307	3.285

**Table 4:** Thermodynamics and kinetics parameters for the epoxidation of seed oil extract from melon

Temperature	Rate constant K (hr <sup>-1</sup> )	1/T	$k \text{ h}/k_B T$ (x 10 <sup>-15</sup> )	$\ln [k \text{ h}/k_B T]$
30°C (303K)	0.032	0.0033	5.07	-32.92
50°C (323K)	0.041	0.0031	6.09	-32.73
60°C (333K)	0.059	0.0030	8.51	-32.40
70°C (343K)	0.24	0.0029	33.6	-32.02

### Kinetics and thermodynamics of the epoxidation of seed oil extract from melon

The in situ epoxidation reaction process, kinetic reaction steps, expected kinetic rate equation and profile for the epoxidation of unsaturated fatty acid methyl esters have been reported by Shuangfei and Wang, (2011) and adequately described by Akpoveta and Adetunji, (2022) in the kinetics study of *in situ* epoxidation of *Allanblackia floribunda* seed oil, since the epoxidation process for melon seed oil follows the same kinetic mechanism. The rate equation was applied;  $d C_{\text{ep}}/dt = k (C_{\text{hpo}} - C_{\text{ep}})$ . Caa<sub>0</sub> where  $k$  is the rate constant,  $C$  represents the concentration of different components; subscripts hp<sub>0</sub>, aa<sub>0</sub> and ep represents the initial concentrations of H<sub>2</sub>O<sub>2</sub>, CH<sub>3</sub>COOH and epoxides respectively. The integrated form of the rate equation above is given as;  $\ln(C_{\text{hpo}} - C_{\text{ep}}) = -K \cdot Caa_0 \cdot t + \ln C_{\text{hp}}$  The plot of  $\ln(C_{\text{hpo}} - C_{\text{ep}})$  vs. time should yield straight lines for reactions with negligible degradation of oxirane, with a slope of  $-K$  for a pseudo zero order kinetic mechanism.

Pseudo zero order kinetics was observed for the epoxidation processes at the different temperatures studied (30°C-70°C) from the kinetic profiles in Figures 2-5. This is with respect to oxirane formation in the presence of high hydrogen peroxide (30%) concentration when compared to the percentage oxirane content formed. The plots of  $\ln(C_{\text{hpo}} - C_{\text{ep}})$  vs. time for all the temperatures studied (30°C-70°C) yielded straight lines as seen in Figures 2-5. Linearity in the plots as seen in the near unity values of their regression coefficients ( $R^2 > 0.9$ ) accounts for their pseudo zero order kinetics. Similar kinetics have been reported in the kinetics study of *in situ* epoxidation of *Allanblackia floribunda* seed oil (Akpoveta and Adetunji, 2022); *Jatropha* seed oil using peroxyacetic acid (Okiemen and Okiemen, 2001), and that for the epoxidation of unsaturated fatty acid methyl esters in the presence of SO<sub>3</sub>H functional Brønsted Acidic ionic liquid as catalyst (Shuangfei and Wang, 2011).

Their rate constants were obtained from the slope of the linear plots and found to be 0.032hr<sup>-1</sup>, 0.041hr<sup>-1</sup>, 0.059hr<sup>-1</sup> and 0.24hr<sup>-1</sup> for the

epoxidation process at 30°C, 50°C, 60°C and 70°C respectively. In the case of deviation from linearity of increasing oxirane content at higher temperature of 70 °C, the rate constant was obtained from the initial slope. The rate constants shows an increase in epoxidation rate with increasing temperature, thereby justifying the highest oxirane content of 5.87% obtained at 70°C. The reports of Okiemen and Okiemen (2001), Shuangfei and Wang (2011), Akpoveta and Adetunji (2022); agrees and supports this trend. Thermodynamic parameters such as activation energy ( $E_a$ ), activation enthalpy ( $\Delta H^\ddagger$ ), gibb's free energy of activation ( $\Delta G^\ddagger$ ) and activation entropy ( $\Delta S^\ddagger$ ) for the epoxidation process were all determined from the data presented in Table 4. The average value of activation energy ( $E_a$ ) is determined from the Arrhenius equation below, using the temperature dependence of the reaction rate constants.

$$\log \frac{k_2}{k_1} = \frac{E_a}{2.303R} \left( \frac{T_2 - T_1}{T_2 T_1} \right)$$

The activation energy is calculated to be 133.11 kJmol<sup>-1</sup>. This is seen to be higher than the values of 33.73 kJmol<sup>-1</sup>, 65.66 kJmol<sup>-1</sup>, 65.6 kJmol<sup>-1</sup>, and 65.57 kJmol<sup>-1</sup>, reported for the epoxidation of *Allanblackia floribunda* seed oil (Akpoveta and Adetunji, 2022), rubber seed oil (Aigbodion *et al.*, 1999), unsaturated fatty acid methyl esters (Shuangfei and Wang, 2011) and *Jatropha* seed oil (Okiemen and Okiemen, 2001). High activation energy when compared to the others reported in literature indicates a greater temperature dependence of the reaction rate and this explains why the epoxidation proceeds faster at higher temperatures. The Eyring equation below was employed in the determination of enthalpy of activation ( $\Delta H^\ddagger$ ) and entropy of activation ( $\Delta S^\ddagger$ ).

$$\ln \frac{k h}{k_B T} = \frac{\Delta S^\ddagger}{R} - \frac{\Delta H^\ddagger}{RT}$$

An Eyring plot of  $\ln(kh/k_B T)$  versus  $1/T$  gives a line with slope of  $-\Delta H^\ddagger/R$  and intercept of  $\Delta S^\ddagger/R$  as seen in Figure 6.

The free energy of activation is determined using the Gibb's thermodynamics equation  $\Delta G^\ddagger = \Delta H^\ddagger - T\Delta S^\ddagger$ .

The activation enthalpy ( $\Delta H^\ddagger$ ), activation entropy ( $\Delta S^\ddagger$ ) and Gibb's free energy of activation ( $\Delta G^\ddagger$ ) were calculated to be 18.22 kJmol<sup>-1</sup>, -0.21 kJmol<sup>-1</sup> K<sup>-1</sup> and 87.95 kJmol<sup>-1</sup> respectively. The values were slightly higher than those reported ( $\Delta H^\ddagger = 17.04$  kJmol<sup>-1</sup>  $\Delta S^\ddagger = -0.224$  kJmol<sup>-1</sup> K<sup>-1</sup>) for the epoxidation of *Allanblackia floribunda* seed oil (Akpoveta and Adetunji, 2022), except for Gibb's free energy of activation ( $\Delta G^\ddagger = 91.632$  kJmol<sup>-1</sup>). The activation enthalpy ( $\Delta H^\ddagger$ ), activation entropy ( $\Delta S^\ddagger$ ) and Gibb's free energy of activation ( $\Delta G^\ddagger$ ) values were however lower when compared to the report of Shuangfei and Wang (2011) for unsaturated fatty acid methyl esters epoxidation process ( $\Delta H^\ddagger = 62.8$  kJmol<sup>-1</sup>,  $\Delta S^\ddagger = -0.19$  kJmol<sup>-1</sup> K<sup>-1</sup>,  $\Delta G^\ddagger = 127.6$  kJmol<sup>-1</sup>). They were also below the reported values ( $\Delta H^\ddagger = 64.73$  kJmol<sup>-1</sup>,  $\Delta S^\ddagger = -0.128$  kJmol<sup>-1</sup> K<sup>-1</sup>,  $\Delta G^\ddagger = 102.63$  kJmol<sup>-1</sup>) for *Jatropha* seed oil epoxidation process (Okiemen and Okiemen 2001). The epoxidation process is endothermic as indicated in its activation enthalpy ( $\Delta H^\ddagger$ ) value of 18.22 kJmol<sup>-1</sup>. The negative activation entropy ( $\Delta S^\ddagger$ ) value of -0.21 kJmol<sup>-1</sup> K<sup>-1</sup> and positive Gibb's free energy ( $\Delta G^\ddagger$ ) value of 87.95 kJmol<sup>-1</sup> clearly shows that the epoxidation process is non-spontaneous. The endothermicity and non-spontaneity of the epoxidation have similarly been observed in the reports of Okiemen and Okiemen (2001), Shuangfei and Wang (2011), and Akpoveta and Adetunji (2022) for different seed oils. The thermodynamic data shows that the epoxidation process is unfavorable for both enthalpy (since  $\Delta H^\ddagger > 0$ ), entropy (since  $\Delta S^\ddagger < 0$ ) and Gibb's energy (since  $\Delta G^\ddagger > 0$ ).

## Conclusion

Oil from melon seeds is seen to be epoxidizable from the finding of this study. The finding reveals optimum epoxidation with minimum oxirane cleavage at temperature of around 70°C; with the formation of oxirane ring not favoured optimally at lower temperatures below 70°C. It could therefore be inferred that the optimal temperature and time required for efficient epoxidation of melon seed oil should be around 70°C at a maximum time of one hour, as this gives relatively high yield of oxirane content without a post hydroxylation reaction which further leads to oxirane ring cleavage. The optimum reaction conditions established from the findings of this work will help in obtaining maximum oxirane yield and saving cost significantly in epoxidation processes for melon seed oil employed in large scale industrial applications.

**Conflicts of Interest:** None

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