

Green polyols from tamanu seed oil: Reaction kinetics and process optimization

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Abstract

Using methanol, this study examined the hydroxylation process of epoxidized tamanu seed oil (ETSO), with an oxirane number of 3.92 to 4.04 mmol/g, under the catalyzation of sulfuric acid (H_2SO_4). The objectives of this study were, first, to synthesize polyol from ETSO, and, second, determine how temperature and catalyst concentration play a role in the hydroxylation process. During the experiment, a second-order reaction kinetic model was used for analysis. The hydroxylation process was conducted in a batch reactor for 4 hours under constant temperatures and stirring speed. During the experiment, the samples were taken every 30 minutes. The oxirane number of ETSO and the concentration of polyols were used to the reaction rates. The optimal conditions were found at a temperature of 65°C, with a methanol-to-epoxide mole ratio of 4:1 and a catalyst concentration of 3%. The pre-exponential factor (A) and the calculated activation energy (E_a) were found to be 59,041.74 g.mmol⁻¹.min⁻¹ and 44.69 kJ/mol, respectively. This research, therefore, has successfully identified the optimal conditions for the synthesis of bio-based polyols from tamanu oil.

Keywords: hydroxylation; mole ratio; polyol concentration; oxirane number; temperature

1. Introduction

Plant diversity worldwide provides both for edible and inedible vegetable oils for human needs. Most edible vegetable oils, including those obtained from soybeans, sesame, sunflower, and canola are usually rich in fatty acids and triglycerides. For their properties, they have been commonly applied in food industries [1,2]. Triglycerides and fatty acids, having been utilized in therapeutic formulations for health improvement, including disease prevention [3], are also applicable in bio-based products for the oleochemical industry [4]. Fatty acids that are commonly found in vegetable oils include myristic, caprylic, linoleic, oleic, lauric, palmitic, stearic, palmitoleic, and linolenic acids. Nowadays, along with the advancement in technology and science, vegetable oils are applicable in various fields, including nutrient-dense foods [2,5,6], biodiesel fuel [7–10], pharmaceuticals [3,11], cosmetics [12,13], and other chemical industries [14]. Following the results of various research and development, they are also commonly used as the primary raw materials in the production of structured triglycerides, also known as structured lipids [15–18].

Tamanu seed kernels contain approximately 75% oil.

Traditionally, oil extracted from tamanu nuts has been topically administered to skin and mucous membrane sores, with the aim at infection prevention and even scars lessening [19]. The fatty acid profile of tamanu oil, averaged from five samples, includes stearic acid (30.2 ± 4.36%), linoleic acid (25.5 ± 3.87%), oleic acid (23.6 ± 4.77%), palmitic acid (16.5 ± 1.59%), docosadienoic acid (1.4 ± 5.08%), arachidonic acid (0.6 ± 0.09%), gadoleic acid (0.3 ± 0.1%), palmitoleic acid (0.26 ± 0.11%), alpha-linoleic acid (0.26 ± 0.05%), behenic acid (0.1 ± 0.15%), and dihomo-gamma-linolenic acid (< 0.1%). Saturated fatty acids make up the majority (41–52%), with stearic acid being particularly abundant (25–35%) [20].

Hydroxylation denotes the introduction of hydroxyl groups into chemical compounds. Vegetable oils can be transformed into their hydroxy derivatives through several routes: ring-opening reaction involving alcohols, amino alcohols, or acids, hydrochlorination, or hydrobromination and catalytic hydrogenation [21–23]. Hydroxylation, in the absence of solvents, can be conducted using an acidic catalyst. Common catalysts include mineral acids, metals, and metal carbonyl compounds. Recently, polyols based on vegetable oils produced through the epoxy ring opening reaction were widely reported. This process consists of two key sequential steps: epoxidizing unsaturated fatty acids in vegetable oils and, followed by, the nucleophilic ring-opening reaction of epoxides with the help of reagents like alcohols, carboxylic acids, or halogenated acids. Alcohol and epoxide, when being left to

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react to each other, can produce β -alkoxy alcohol [24,25]. This reaction can be promoted by acidic and alkaline catalysts, such as porphyrin complexes [26], Lewis's acid [27], and Brønsted acid [24]. According to Petrovic *et al.* (2010), the optimal reaction temperature is approximately 50°C, with a mole epoxide to hydroxyl group ratio ranging from 1:1 to 10:1 [28].

Several studies have investigated the hydroxylation of various epoxidized vegetable oils [28–33], such as those soybean, perilla, and tung oils. Among those producing edible vegetable oil, tamanu contains distinctive chemical features. Yet, no study has focused on tamanu oil in hydroxylation and kinetic studies. Tamanu oil has been found to contain fatty acids, with irregular distribution, including calophyllolide and other resinous compounds. Interestingly, a relatively high content of unsaturated chains that differs structurally from the typical linoleic-oleic profile found in more conventional oils is also discovered in tamanu. These structural features may affect tamanu oil epoxidation efficiency, oxirane stability, and reactivity during alcohol-based ring-opening, which potentially result in kinetic behavior that is not directly comparable to previously reported systems.

A prior literature study revealed that tamanu seed oil has not yet been used as a raw material for hydroxylation reactions. The hydroxylation kinetics or process-variable effects have not been studied, especially catalyst concentration and temperature. Several studies investigated hydroxylation rate constants involving alcohol and epoxy groups as side reactions in vegetable oil epoxidation [34,35]. No analysis of tamanu seed oil was done. Examination the hydroxylation of epoxidized tamanu seed oil (ETSO) is expected to provide an understanding of how its distinct molecular composition impacts reaction pathways and kinetic parameters. This study, therefore, evaluates the influence of catalyst concentration and temperature, and develops the hydroxylation kinetics, for the hydroxylation of ETSO.

2. Materials and Methods

2.1. Materials

The product yielded from tamanu seed oil epoxidation was epoxidized tamanu seed oil (ETSO) [36]. The resulting product had an initial oxirane number of 2.933 mmol/g, with a polyol concentration of 0.11 mmol/g. The chemicals used during the process and analysis included sulfuric acid (98 wt%), acetic anhydride, glacial acetic acid, pyridine, methanol, crystal violet indicator, hydrobromic acid (47 wt%), potassium hydroxide, and potassium hydrogen phthalate.

2.2. Hydroxylation process

In this study, epoxidized tamanu seed oil (ETSO) was produced through the oxidation of tamanu seed oil. Peracetic acid, generated in situ, played as the oxidant, with sulfuric acid acting as the catalyst. The epoxidation process of tamanu seed oil was conducted at 60°C for four hours with a constant stirring rate of 600 rpm [36,37]. To hydroxylate epoxidized tamanu seed oil (ETSO), methanol and 3% sulfuric acid (relative to the total solution) were mixed in an Erlenmeyer flask. In the reaction, up to 70 g of epoxidized tamanu seed oil (ETSO),

mixed with methanol-sulfuric acid solution was involved. The hydroxylation process was carried out under isothermal conditions at temperatures of 45, 55, 65, and 75°C and a stirring rate of 600 rpm. The reaction time was four hours, with samples collected every 30 minutes. The samples were then cooled and separated to isolate the polyol, which was further analyzed for the oxirane number and concentration. Analytical methods were adapted from previous studies [38–40].

3. Results and Discussion

3.1. Effect of temperature on oxirane number and polyol's concentration

Fig. 1(a) illustrates the relationship of the oxirane concentration with reaction time, with sulfuric acid used as a catalyst, at various temperatures. It can be observed that the epoxide concentration, which is expressed by the oxirane number, decreases with increased reaction time at all reaction temperatures. The longer the reaction times (LRT), the more the epoxy groups available to react. This allows the reduction of the residual epoxy content present in the system. Similarly, when the temperature increases, the epoxide concentration reduces over the same reaction time. The greater kinetic energy and activity of reactant molecules at elevated temperatures can offer a reasonable explanation for this phenomenon.

At a temperature of 40°C, the epoxide concentration was found to be relatively steady throughout the process. This reflects a slower rate of reaction. The initial rate of decrease in concentration became faster when the temperature was set to be higher, before slowing down towards the end. This phenomenon clearly indicates that temperature plays a significant role in hydroxylation rate; higher temperature can foster the dynamics of molecules, thus increasing the collision frequency and the kinetic rate of the reaction [32,33,41].

At minute 0, when the process was ready to start, the oxirane concentration appeared to be constant at 2.9333 mmol/g. After 30 minutes, a significant decline occurred. At 70°C, which was set as the highest temperature, the oxirane number fell to 1.9473 mmol/g, and then remained above 2 mmol/g at lower temperatures. It fell to only 2.6773 mmol/g at a temperature of 40°C. After 240 minutes, the lowest oxirane number read 0.5881 mmol/g at a temperature of 70°C. These data indicate that the reaction of hydroxylation works more effectively at higher temperatures. This can be linked to higher kinetic energy of reactants, which can lead to a faster reaction and better results.

Methanol, a primary alcohol, has been known to have a strong nucleophilic effect. Studies reported that hydroxylation of epoxy compounds with primary alcohols usually give higher conversions compared with that using secondary and tertiary alcohols [33,41]. The opening of epoxy rings with secondary alcohols usually needs more LRT. This causes the conversion rates to be lower than those found in earlier studies. For example, the conversion rate of styrene epoxide hydroxylation using methanol and a graphite oxide as the catalyst reaches approximately 94%. The differences in types of raw materials and catalysts used are thought to be the primary cause.

Fig. 1(b) shows the dependence of polyol concentration on the reaction time at various temperatures. Longer reaction time

(LRT) leads to a continuous increase in polyol concentrations for all temperature ranges. It increases the interactions and collisions of particles, thus accelerating the reaction rate. At constant reaction time, polyol concentration continues to increase along with the increase in temperature. Higher temperatures can increase the intensity of molecular movement, which consequently result in more frequent and successful collisions among reactant particles for accelerating the reaction rate.

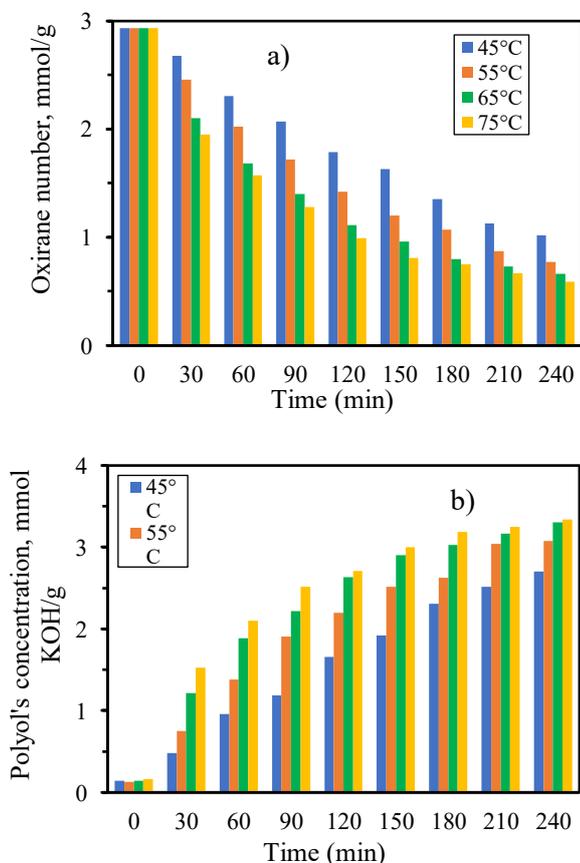


Fig. 1. Oxirane number and polyol concentration at various temperatures

The polyol concentrations obtained at temperatures of 40, 55, 65, and 75°C at minute 240 of reaction are 2.70, 3.07, 3.31, and 3.34 mmol KOH/g, respectively. These figures are similar to those found in previous works: 3.21 mmol KOH/g [42] and 3.40 ± 0.27 mmol KOH/g [43]. Dai *et al.* in their study using different alcohols: 1,2-ethanediol and 1,2-propanediol obtained polyol concentrations of 4.51 mmol KOH/g and 5.16 mmol KOH/g, respectively. Based on these data, it can be concluded that alcohol plays a determining role in the hydroxylation process. Longer alcohol molecules tend to increase the polyol concentrations. However, secondary or tertiary alcohols present steric hindrance that hinders the reaction mechanism.

3.2. Effect of methanol-epoxy mole ratio on oxirane number and polyol concentration

The ratio of methanol to oil ratio determines the reaction mechanism of a hydroxylation process. Methanol ratio, when increases, enhances the availability of protons, thus encouraging the formation of hydroxy intermediates. A correct concentration of oil is needed to provide adequate substrate to

react with methanol; improper adjustment of methanol concentration can inhibit the reaction or cause the formation of by-products. Optimization of these conditions is, therefore, important when the aim is to obtain the best possible results of hydroxylation. In this study, the methanol-to-epoxy mole ratios tested were 1:1, 2:1, 4:1, and 5:1.

Fig. 2(a) illustrates that both reaction time and methanol-to-epoxy mole ratio significantly exhibit their effects on the concentration of polyols. The initial concentration of polyols when the process began, namely at minute 0, was low and varied between 0.1309 mmol KOH/g and 0.1419 mmol KOH/g. At minute 30 of reaction, a considerable increase, especially for the 4:1 ratio, which reached 1.1557 mmol KOH/g, was observed. Further increases were recorded until 240 minutes when the highest polyol concentration was 3.1527 mmol KOH/g at the 4:1 ratio. This indicates that hydroxylation reached its best at this ratio. At this methanol-to-epoxy mole ratio, the reactants appeared to be almost ideal proportions for effective interaction. The hydroxylation reaction worked more effectively at higher methanol-to-epoxy ratios. These findings were in line with previous studies, which revealed that increasing reactant components could enhance the formation of the reaction products [41,44]. Therefore, this study highlights that selecting the optimal methanol-to-epoxy ratio to maximize polyol production from tamanu seed oil is a substantial step.

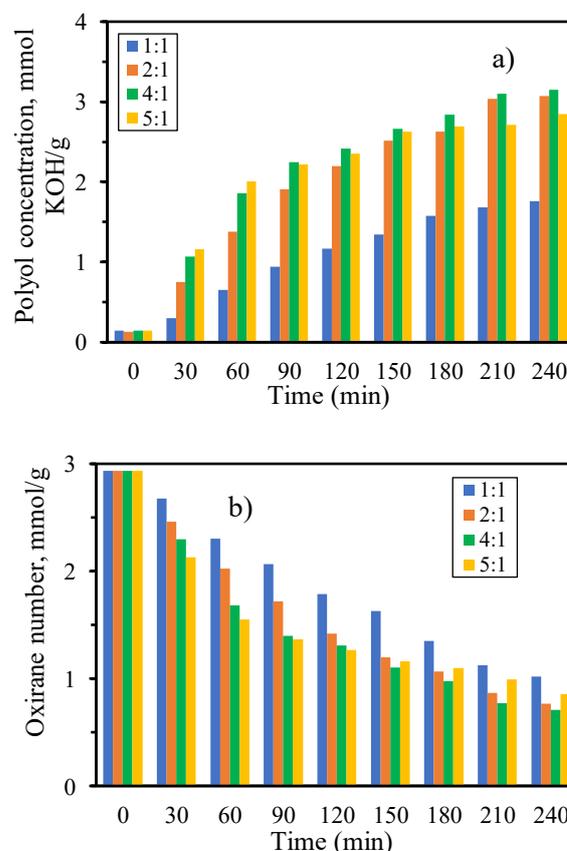


Fig. 2. Effect of mole ratio on polyol concentration and oxirane number in the hydroxylation process

The product decreased at the ratio of 5:1, where the highest polyol concentration reached only 2.85 mmol KOH/g at minute 240. Excessive methanol hampered the reaction and slowed the formation of hydroxy intermediates. Furthermore, kinetics and

thermodynamic limitations declined the concentration of polyol when the methanol-to-epoxy mole ratio was excessive (5:1 is considered excessive). Excessive methanol could also dilute the reactive epoxy groups, leading to the reduction of the effective collision frequency between epoxide rings and methanol molecules. Therefore, the probability of nucleophilic attacks reduced, slowing down the hydroxy intermediate formation rate.

Fig. 2(b) depicts a notable decline in the oxirane number over time and with varying methanol-epoxy mole ratios. At minute 0 across all ratios, the oxirane number was constant at 2.9333 mmol/g. At minute 30, at a 1:4 ratio, the oxirane number dropped to 2.1273 mmol/g. The decline was observed to be continue until minute 240. The oxirane number appeared to reach its lowest value of 0.7681 mmol/g at the ratio of 1:4. The hydroxylation process was found to be more effective in consuming oxirane at higher epoxy proportions. This finding was in agreement with those of a study by Yelwa *et al.*'s (2019), which revealed that increasing the epoxy content can increase conversion efficiency and lower oxirane numbers. The reduction in oxirane number was considered significant, as it indicated a more thorough reaction and more stable hydroxylation products [44].

Fig. 2(b) shows that the progressive acid-catalyzed ring-opening of epoxy groups by methanol can decline oxirane number. H_2SO_4 , which protonates the oxirane ring to increase its electrophilicity, increases the possibility of nucleophilic attacks [45]. The reaction rate tends to be higher when the methanol-to-epoxy ratio increases. This is due to the mass transfer between the polar methanol phase and the nonpolar oil phase, which increases the contact between the reactants. Methanol, which is a small molecule, also reduces steric hindrance to the access of protonated oxirane sites, compared to larger alcohols. Meanwhile, intramolecular steric constraints within the triglyceride chains make some oxirane groups react more slowly. This can lead to the gradual decline over time. Greater availability of protonated oxirane groups, indicated by higher epoxy contents, has been reported to enhance hydroxylation efficiency [44]. This phenomenon leads to the more rapid decline at the 1:4 ratio. Both mechanistic and physicochemical factors explain why the consumption of oxirane varies across time and mole ratio, beyond the numerical trends exhibited.

3.3. Effect of catalyst concentration

To investigate the influence of catalyst concentration on epoxidized tamanu seed oil (ETSO) hydroxylation, the reaction was conducted at 60°C with a 600-rpm stirring. The mole ratio of methanol to epoxidized tamanu seed oil (ETSO) was maintained at 1:1. Sulfuric acid, being the catalyst of the reaction, enabled methanol to act as a nucleophile and further react with epoxidized tamanu seed oil (ETSO). One of the most important factors that can accelerate the reaction is the catalyst concentration. However, excessive concentration can burn or change the color of the resulting polyol. For this reason, the catalyst concentration in this study was set to be 1%, 2%, 3%, and 5% during the experiment to determine the optimum one.

Table 1 presents the relationship between oxirane number, reaction time, and catalyst concentration. As mentioned earlier,

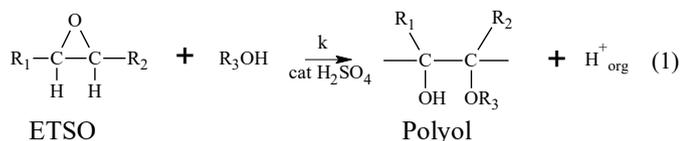
increased reaction time can decrease epoxy concentration because longer reaction times lead to more collisions and interactions between the reactants, which in turn decreases the epoxy concentration due to the conversion to hydroxyl or polyol compounds. Generally, the relationship between catalyst concentration and oxirane number could be inversely related. When the catalyst concentration increases, more oxirane molecules react with methanol. The catalyst incorporated helps reduce the activation energy, thus increasing the reaction rate. However, H_2SO_4 , when used as the reaction catalyst at concentrations $\geq 5\%$, turns the polyol dark brown. Such a change can also result in the product denaturation, thereby decreasing the quality of the resulting product. These data indicate that a catalyst concentration of 3% is optimal for the specified reaction. This figure is slightly different from Yadav *et al.*'s finding: 5% [46]. This difference is likely due to the catalyst type used, particularly $Fe(Cp)_2BF_4$.

Table 1. The correlation between oxirane number with time and catalyst concentration

<i>t</i> , min	Oxirane Number			
	Cat 1%	Cat 2%	Cat 3%	Cat 5%
0	2.9333	2.9333	2.9333	2.9333
30	2.4588	2.1214	1.8372	1.6632
60	2.0057	1.7054	1.3469	1.1527
90	1.6915	1.3895	1.1163	0.8805
120	1.4404	1.1762	0.9425	0.8211
150	1.2172	1.0230	0.8352	0.7204
180	1.0608	0.8540	0.7205	0.6421
210	0.8517	0.7686	0.6429	0.5747
240	0.7162	0.6615	0.5768	0.5249

3.4. Kinetics of hydroxylation process

The hydroxylation kinetics can be used to assess the second-order reaction model. This study proposed two presumptions (1) no adverse reaction occurs and (2) hydroxylation occurs as a pseudo-homogeneous reaction. The first assumption was made based on the natures of methanol and epoxidized tamanu seed oil (ETSO) as organic substances that dissolve well in one another. Sulphuric acid is an inorganic substance that is only weakly soluble in these organic substances; its content is negligible (i.e., 3%). Therefore, in general, the reaction can be regarded as pseudo-homogeneous. The second assumption was made based on experimental findings, which showed that the concentration of polyol generated was directly proportional to the concentration of oxirane that reacts. Epoxidized tamanu seed oil (ETSO) and methanol had an equimolar molar ratio of 1:1. Three percent of the reactants' total weight was made up of H_2SO_4 . The discussion in section 3.3, concluding that 3% is the ideal catalyst concentration, served as the basis for the selection of the catalyst concentration used. For four hours, the procedure was performed in a batch reactor. The reaction occurred isothermally under a 600-rpm constant stirring. Eq. (1) illustrates the epoxidized tamanu seed oil (ETSO) hydroxylation mechanism.



According to the elementary reaction law, each reactant's concentration determines the rate of a reaction. Eq. (2) can be used to represent the reaction rate.

$$\frac{d[\text{polyol}]}{dt} = k[\text{R}_3\text{OH}][\text{ETSO}] \quad (2)$$

where [polyol] is polyol's concentration, k represents rate constant, [R₃OH] is methanol concentration, and [ETSO] stands for epoxidized tamanu seed oil concentration.

[R₃OH] = [ETSO] due to Equi molarity ([R₃OH]₀ = [ETSO]₀) and the fact that the coefficient of R₃OH is equal to the coefficient of epoxy (ETSO). Eq. (2) can be transformed into Eq. (3).

$$\frac{d[\text{polyol}]}{dt} = k[\text{ETSO}]^2 \quad (3)$$

Eq. (3) shows that hydroxylation has a second-order reaction. Based on this, Eq. (4) is then used to calculate the sum of squared errors (SSE) of this kinetics model.

$$\text{SSE} = \sum \{[\text{polyol}]_{\text{data}} - [\text{polyol}]_{\text{calc}}\}^2 + \sum \{[\text{ETSO}]_{\text{data}} - [\text{ETSO}]_{\text{calc}}\}^2 \quad (4)$$

where SSE is the sum of squared errors, [polyol]_{data} and [ETSO]_{data} are the experimental concentration of polyol and epoxidized tamanu seed oil, respectively. Then, [polyol]_{calc} and [ETSO]_{calc} are the calculated concentration of polyol and epoxidized tamanu seed oil, respectively.

The epoxidized tamanu seed oil (ETSO) concentration and polyol concentration for each temperature were the experimental data used in this model. By using SSE as the objective function, the kinetic parameter (k) was determined. Fig. 3 displays the algorithm used to calculate the reaction rate constant.

Table 2 displays the SSE value and the reaction rate constant for each temperature. The reaction order of the reaction rate constant is 10⁻³ g.mmol⁻¹.min⁻¹, consistent with the earlier research's finding [34]. The reaction temperature and the reaction rate constant have a direct relationship. For the 45–75°C temperature range, the k values range from 2.657 × 10⁻³ to 11.270 × 10⁻³ g.mmol⁻¹.min⁻¹. At 65°C and 75°C, the lowest SSE values are reported for epoxy and polyol concentrations. Furthermore, with an average SSE of 0.100, the sample that was processed at a temperature of 45°C produced the highest value of SSE. Table 2 shows that the average SSE values drop at temperatures between 45°C and 65°C. However, the SSE average values increase once more at 75°C, suggesting that the side reactions might have started at this temperature. While epoxy's SSE values range from 1.151 to 9.857, those for polyol concentrations range from 0.004 to 1.206, implying that the model's outputs match to experimental data regarding epoxy and polyol concentrations. Overall, the evaluated kinetics model can be classified as satisfactory.

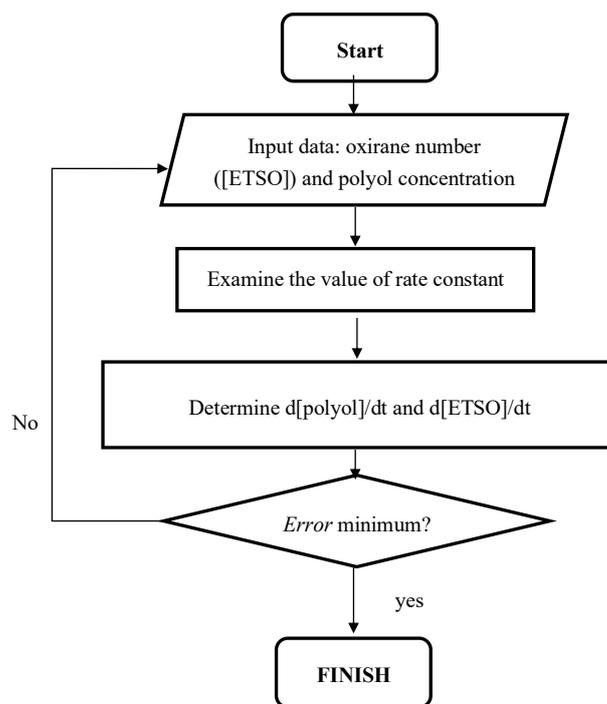


Fig. 3. Reaction rate constant (k) calculation algorithm

Table 2. Values for the reaction rate constant (k) and SSE in the hydroxylation kinetics model

T (°C)	T (K)	$k \times 10^3$ (g.mmol ⁻¹ .min ⁻¹)	SSE		
			ETSO	Polyol	Average
45	318.15	2.657	9.857	1.206	5.532
55	328.15	4.602	1.402	1.126	1.264
65	338.15	7.671	1.151	0.004	0.578
75	348.15	11.270	1.173	0.009	0.591

Although fitting a second-order kinetic model successfully describes epoxidized tamanu seed oil (ETSO) hydroxylation, this approach should be recognized to have several inherent limitations. The model is based on a pseudo-homogeneous system and neglects mass-transfer resistance. The paper describes that high viscosity of epoxidized tamanu seed oil (ETSO) (especially at 40°C) causes poor initial mixing and results in larger deviations of experimental and modeled epoxy concentrations during the first 30 minutes [33]. This behavior indicates that the early reaction stage can be affected by diffusion or mixing limitations. Therefore, the measured rate constant may reflect an apparent rate rather than a purely intrinsic chemical rate. This observation is consistent with previous kinetic studies of epoxidized vegetable oils; viscosity, phase behavior, and mixing efficiency affect the quality of second-order or pseudo-first order kinetic fits [34,35,47].

Using the Arrhenius equation, the activation energy (E_a) and pre-exponential factor (A) can be calculated using the reaction rate constants obtained for each temperature. The relative error value of the curve is considered low, while the R -squared is considered satisfactory, as its value is 0.9973. The curve slope can be assessed through the $-E_a/R$ value, in which R represents the ideal gas constant. Meanwhile, the intercept curve can be determined by considering the $\ln(A)$ value. The pre-exponential factor (A) and the calculated activation energy

(*E_a*) are 59,041.74 g.mmol⁻¹.min⁻¹ and 44.69 kJ/mol, respectively. This study's findings are slightly different from earlier studies on perilla and soybean oils: 29.56 and 39.99 kJ/mol [42] and 43.11 kJ/mol [48], respectively.

The activation energies obtained in this study are slightly higher than those reported for soybean and perilla oils, but the difference is still within a reasonable range. This finding suggests that tamanu seed oil may have diffusion characteristics or structural barriers that are not entirely similar to those of these two oils. Although the second-order model provides a reasonably good numerical fit, potential transport effects still need to be considered. Comparing these results with previous kinetic studies provides a stronger interpretation, as the results can be understood as apparent kinetics influenced by a combination of chemical and physical factors.

4. Conclusion

The hydroxylation reaction with sulfuric acid as a catalyst affects epoxide and polyol concentrations significantly during different temperatures. LRT resulted in the reduction of epoxy resin. Higher temperatures accelerated the reaction. Following 240 minutes of reaction, the polyol concentrations at 45°C, 55°C, 65°C, and 75°C were 2.70, 3.07, 3.31, and 3.34 mmol KOH/g, respectively. Optimal production of polyol depends on both the methanol-to-epoxy ratio and oil concentration. A higher methanol-epoxy ratio increases the amount of polyol produced with the reduction of oxirane numbers. However, at a ratio of 5:1, the yield decreased and the maximum recorded polyol concentration was approximately 2.85 mmol KOH/g at 240 minutes. The study also evaluated the catalyst concentration within this hydroxylation of epoxidized tamanu seed oil by adjusting the concentration of sulfuric acid, H₂SO₄, from 1 to 5% to reach the optimal percentage. It was concluded that the optimal catalyst concentration was 3%, which is slightly lower than that used in the work of Yadav *et al.* at a concentration of 5%, probably due to the different catalyst types used. For the 45–75°C temperature range, the *k* values were 2.657 x10⁻³ to 11.270 x10⁻³ g.mmol⁻¹.min⁻¹. At 65°C and 75°C, the lowest SSE values were recorded for epoxy and polyol concentrations.

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