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Synthesis and characterization of polyols made from poultry fat

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ABSTRACT

Objective: Identify the optimal conditions of polyols synthesis from poultry fat by epoxidation and hydrolysis *in situ*.

Design/methodology/approach: Using a statistical design of response surface Box-Behnken type was evaluated the effect of the factors temperature (60, 70 and 80 °C), catalyst (1, 2 and 3 % w/w), molar ratio double bonds:acetic acid (1: 1, 1: 1.5 and 1: 2) and time (4, 6 and 8 h), on the acid index of polyols synthesized.

Results: The FTIR spectra indicated that in mild reaction conditions epoxide groups (827 cm⁻¹) are generated while in severe reaction conditions the formation of OH groups is favored. Therefore, the optimal conditions for the generation of polyol were: 80 °C, 3% w/w catalyst (H₂SO₄), molar ratio double bonds:acetic acid 1:2 and reaction time of 8 h, obtaining a maximum percentage of 78% acidity index, and hydroxyl number of 74 mg KOH/g.

Limitations on study/implications: The removal of the acid medium and water in the reaction was a challenge in polyols with higher acidity index.

Findings/conclusions: The method used of fat extraction, allows to obtain raw material that meets the characteristics to perform the reaction of epoxidation and hydrolysis in a single step to obtain polyol. The most severe conditions of temperature, catalyst concentration, molar ratio of double bonds: acetic acid and reaction time, allowed to obtain the greatest amount of polyol from poultry fat.

Keywords: Hydrolysis, animal fat, epoxidation, agro-industrial waste.

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INTRODUCTION

Recently, there has been an increased interest in renewable raw materials to produce biodegradable materials (Saalah *et al.*, 2017). Polyols are precursors to different polymers, especially polyethers, polyesters, and polyurethanes (Ionescu *et al.*, 2016). However, most commercial polyols are derived from petrochemicals. The most used raw materials for obtaining biopolyols are polysaccharides, carbohydrates, glycerol, and triglycerides, the latter of which can come from either plant or animal sources (Kyriacos, 2020). The most common vegetable oils that have been used for polyol synthesis are soybean (Feng *et al.*, 2017), grapeseed (De Haro *et al.*, 2019), sunflower (Omran *et al.*, 2016), ricinus (Choi *et al.*, 2015), palm (Yeoh *et al.*, 2020), canola (Kong *et al.*, 2007), which have been transformed through reactions such as ozonolysis, transesterification, epoxidation, and oxirane ring opening (Polaczek *et al.*, 2021). Epoxidation reaction is one of the most convenient methods for obtaining polyols from oils due to the presence of carbon-carbon double bonds in triglycerides, where an epoxy group is introduced and through hydrolysis, the hydroxyl group is formed (Purwanto, 2010). The reaction yield depends on temperature, molar

ratio of double bonds:ring-opening reagent, and reaction time. Vegetable oils with a higher degree of unsaturation produce polyols with more hydroxyl groups, resulting in polymers with higher crosslinking density and tensile strength. However, the vegetable oils commonly reported are edible, which generates controversy over their use as raw materials for industrial products. There are reports of triglyceride composition of fish and bovine oils with characteristics suitable for use as raw materials for polymer synthesis (Rohman *et al.*, 2012). In Mexico, more than 3.7 million tons of chicken were produced on 2022 (SIAP, 2022) of which 15% corresponds to fatty residues. Lee and Foglia (2000) mention that chicken fat can be considered a source of unsaturated fatty acids (45-50%), making it attractive for chemical transformations. The objective of this study was to analyze the possibility of using chicken fat for the synthesis of polyols by epoxidation and *in situ* hydrolysis, using a Box-Behnken type response surface statistical design. Four controllable factors were evaluated to achieve maximum conversion: temperature, molar ratio of double bonds:acetic acid, time, and catalyst amount.

MATERIALS AND METHODS

Extraction and characterization of raw material

The chicken fat residues were collected from markets in Tuxtla Gutiérrez, Chiapas, Mexico, solids were removed, and the remaining material was stored at 4 °C until use. Subsequently, the fat was extracted by heating at 90 °C for 6 h. The extracted fat was cooled to room temperature and filtered, cooled, and stored at 4 °C. It was then bleached, deodorized, and fractionated according to the method described by Hernández-Cruz *et al.* (2015). For oxidative stability determination (EN 14112), a biodiesel Rancimat[®] equipment (Metrohm, Herisau, Switzerland) was used, 3 g of sample were heated to 110 °C and 10 L/h air flow. The kinematic viscosity (ASTM D445) was determined with a SVM 3000[®] equipment (Anton Paar, Graz, Austria) at 40 °C and 100 °C. To determine the fatty acid profile, the fat was derivatized by alkaline hydrolysis (100 μ L) to which 1.0 mL of 0.5 M NaOH was added and heated at 80 °C for 20 min with constant agitation. 1.0 mL of BF₃/MeOH 14% was added and kept at 20 °C for 20 min with constant agitation. Afterwards, the methyl esters were extracted with 1.0 mL of hexane. 1 μ L of the extract was injected into a gas chromatograph model 5975 inert XL (Agilent Technologies, Santa Clara, USA), equipped with a DBWax column of 60 m \times 0.25 mm \times 0.25 μ m. The initial temperature in the oven was 150 °C and was maintained for 5 min, then raised to 210 °C (30 °C/min), from 210 °C to 213 °C (1 °C/min), and finally to 225 °C (20 °C/min), it was maintained for 20 min. Helium was used as the carrier gas with a flow of 1 mL/min, with an injector temperature of 250 °C and a split injection ratio of 50:1. An Agilent Technologies model 5975 inert XL mass spectrometer was used to identify the chromatographic signals. The mass spectra were obtained by electron impact ionization at 70 eV. The mass spectra were compared with the HP Chemstation-NIST MS Library version A.00.1995 database. The functional groups were identified by FTIR spectroscopy using a Nicolet[™] iS[™]10 spectrometer (Thermo Scientific, Waltham, USA). Spectra were obtained after 40 scans in a range of 4000 to 500 cm⁻¹ (Jayavani *et al.*, 2017). To obtain the ¹H and ¹³C NMR spectra, a DD2

model NMR equipment from Agilent (California, USA) was used at 500 MHz, 25°C and CDCl_3 as solvent. For the determination of acidity index (AOCS Te 1a-64, 2017), 95% neutral ethanol was heated to near boiling and 0.5 M KOH was added until the endpoint was reached. Five grams of sample were added into a 250 mL Erlenmeyer flask, 85 mL of hot neutral 95% ethanol and 0.5 mL of 1% ethanolic phenolphthalein were added, and it was titrated with 0.5 M KOH until the endpoint was reached. The volume of KOH spent was substituted in the following formula (equation 1):

$$\text{Acidity index} \left(\text{mg} \frac{\text{KOH}}{\text{g}} \text{ of sample} \right) = \frac{V \times M \times 56.11}{P} \quad (1)$$

Where V is the volume of KOH used during titration (mL); M is the molarity of KOH, and P is the mass of the sample (g).

Epoxidation reaction and obtaining polyols

In the epoxidation reaction and obtaining of polyols, acetic acid (HAc) and hydrogen peroxide (H_2O_2) were added in an equimolar ratio with the moles of double bonds present in the chicken fat. To determine the moles of double bonds, the iodine value was used according to the following formula (equation 2):

$$\text{mol}(=) = \frac{IY}{2 \times Mi \times 100} \times P \quad (2)$$

Where $\text{mol}(=)$ are the moles of double bonds; IY is the iodine value obtained, Mi is the molecular weight of iodine (g), and P is the weight of the sample (g). For the synthesis of the polyol, 91.47 g of chicken fat was placed in a flask and the calculated amount of HAc and catalyst (H_2SO_4) was added. The mixture was kept under constant agitation and a temperature below 30 °C. Afterwards, H_2O_2 was added dropwise for 30 minutes, the temperature was increased, and the reaction was kept for the times described in the experimental design. Once the reaction time was reached, 5% NaHCO_3 was added, followed by washing with 10% NaHCO_3 , then water, and finally 5% NaHCO_3 until neutrality was reached. Finally, 20% w/w Na_2SO_4 was added to the organic phase (Salimon *et al.*, 2014).

Experimental design of the epoxidation reaction

To investigate the impact of operational variables on the response, a Box Behnken type design with response surface was conducted. Catalyst (% w/w, X1), acetic acid (mol/mol(=), X2), reaction temperature (°C, X3), and reaction time (h, X4) were chosen as independent variables. The range of values and coded levels of the variables are presented in Table 1. A polynomial equation was used to forecast the response as a function of independent variables and their interactions.

Table 1. Independent variables in the Box-Behnken design for the epoxidation and ring opening reaction of chicken fat. *(%w/w) relative to the sum of acetic acid and H₂O₂.

Factors	Levels			
		-1	0	1
Catalyst (%p/p)*	X ₁	1	2	3
Acetic acid (mol/mol (=))	X ₂	1	1.5	2
Temperature (°C)	X ₃	60	70	80
Time (h)	X ₄	4	6	8

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \sum \beta_{ij} x_{ij}$$

Where β_0 , β_i , β_{ii} and β_{ij} are constant, linear, square and interaction regression coefficient terms, respectively, and x_i and x_j are independent variables. The STATGRAPHICS Centurion XV was used for multiple regression analysis and analysis of variance (ANOVA). The goodness of fit of the model was evaluated by the coefficient of determination R^2 .

Analysis of the epoxidation reaction product and polyol synthesis

To identify the functional groups corresponding to the reaction products, infrared analysis, acidity index, and 1H and 13C nuclear magnetic resonance spectra were performed, as described previously.

Quantification of hydroxyl groups number in polyols

One gram of sample was placed in a 50 mL flask, 10 mL of tetrahydrofuran (THF) was added, mixed, and then 10 mL of catalyst solution (1 g of pyridine in 100 mL of THF) followed by 5 mL of acetylating solution (5 mL of anhydrous acetic acid in 50 mL of THF) were added and stirred for 10 min at 25 °C. Then, 10 mL of hydrolysis solution (20 mL of water in 80 mL of THF) was added and stirred for 30 min. The solution was titrated potentiometrically with 1N ethanolic KOH until pH change. The calculation of the number of hydroxyl groups was obtained using equation 3:

$$ONH = \frac{(V2 - V1 * N * 56.11)}{m} + AN$$

Where ONH is the value of the number of hydroxyl groups; $V2$ is the volume of KOH used in titration of the blank (mL); $V1$ is the volume of KOH used in titration of the sample (mL); N is the normality of KOH; M is the weight of the sample (g), and AN is the acidity index of the sample (Zhang *et al.*, 2015).

RESULTS AND DISCUSSION

Obtaining and characterization of raw material

The extraction yield of chicken fat was 60.71%, similar to that reported by Hernandez-Cruz *et al.* (2020). Additionally, the acidity index was 0.89 mg KOH/g, is similar to that

reported for jatropha oil, although lower than that of castor oil (Hazmi *et al.*, 2013). Considering that the acidity index is defined as the number of mg of KOH required to neutralize acidic hydrogen atoms in 1 g of sample, the higher value of acidity index in castor oil is due to the presence of a hydroxyl group in its chemical structure, which is absent in chicken fat and jatropha oil (Valero *et al.*, 2008). With respect to chicken fat oxidative stability (1.42 ± 0.01 h), it is within the range reported for vegetable oils such as sunflower oil (1.1 h), cottonseed oil (1.9 h), jatropha, and moringa oils (1.1 h) (Murru *et al.*, 2021), oils commonly used for the generation of a wide variety of polymers. The value of oxidative stability is indicative of the presence of double bonds that are oxidized under the conditions in which the determination is made (air flow and high temperatures). In other words, the greater the oxidative stability, the greater the number of double bonds, which are the functional groups required for epoxidation, hydrolysis, and obtaining polyol. With respect to kinematic viscosity ($66.45 \text{ mm}^2/\text{s}$ at $25 \text{ }^\circ\text{C}$, $36.72 \text{ mm}^2/\text{s}$ at $40 \text{ }^\circ\text{C}$, and $8.09 \text{ mm}^2/\text{s}$ at $100 \text{ }^\circ\text{C}$), it is within the range of viscosities of sunflower oil ($64.4 \text{ mm}^2/\text{s}$), olive oil ($74 \text{ mm}^2/\text{s}$), and soybean oil ($60.5 \text{ mm}^2/\text{s}$) (Quinchia *et al.*, 2010), which are renewable raw materials used for the generation of polyols. Saturated fatty acids with carbon chains generate higher viscosities, and the presence of double bonds causes a decrease in viscosity. In relation to the fatty acid profile, monounsaturated fatty acids (47.37%) are observed in greater proportion, with predominance of oleic acid, followed by saturated fatty acids with 33.17% and majority of palmitic acid, and 19.24% of polyunsaturated fatty acids, with linoleic acid being the principal one. The composition of chicken fat allows for the synthesis of epoxides due to the presence of double bonds that will be oxidized and subsequently hydrolyzed. In the FTIR spectroscopy analysis, the stretching band of unsaturated fatty acid $=\text{C}-\text{H}$ is observed at 3006 cm^{-1} , and at 2922.15 cm^{-1} and 2853.36 cm^{-1} corresponding to the stretching of Csp^3-H bonds. In addition, the ester-type carbonyl group band is observed at 1743.20 cm^{-1} , $\text{C}=\text{C}$ band at 1656.40 cm^{-1} , and the saturated chain $\text{C}-\text{H}$ bending vibrations at 1460.36 cm^{-1} and 1372.73 cm^{-1} , as well as the glycerol band at 1157.65 cm^{-1} . The presence of the previously described bands corresponds to the functional groups present in triglycerides, confirming the structure (Wong *et al.*, 2017). The ^1H NMR spectrum of the chicken fat (Figure 1) shows signals at 5.2 and 5.5 ppm of the vinyl protons $-\text{C}=\text{C}-\text{H}$; at 4.1 and 4.3 ppm of the glycerol protons $\text{H}_2-\text{C}-\text{COO}-$; the protons adjacent to the carbonyl group ($-\text{CH}_2-\text{COO}-\text{CH}-$) at 2.7 ppm; at 1.3, 1.6, 2 and 2.4 ppm of the CH_2 groups in the carbon chain, and at 0.8 ppm of the $-\text{CH}_3$ groups.

Epoxidation and hydrolysis reaction

Figure 2 shows the Pareto chart, where it can be observed that all factors have a statistically significant effect on the acidity index and an R^2 of 76.3708.

The response surface plot (Figure 2B) shows a maximum acid value of 78 mg KOH/g, and optimal reaction conditions of temperature at $80 \text{ }^\circ\text{C}$, molar ratio of double bonds to acetic acid at 2, 3% catalyst, and a reaction time of 8 hours, generating the equation from the adjusted model in relation to the response variable:

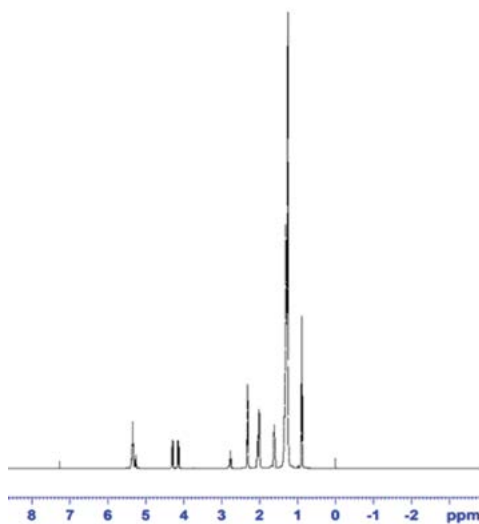


Figure 1. RMN 1H spectrum of chicken fat.

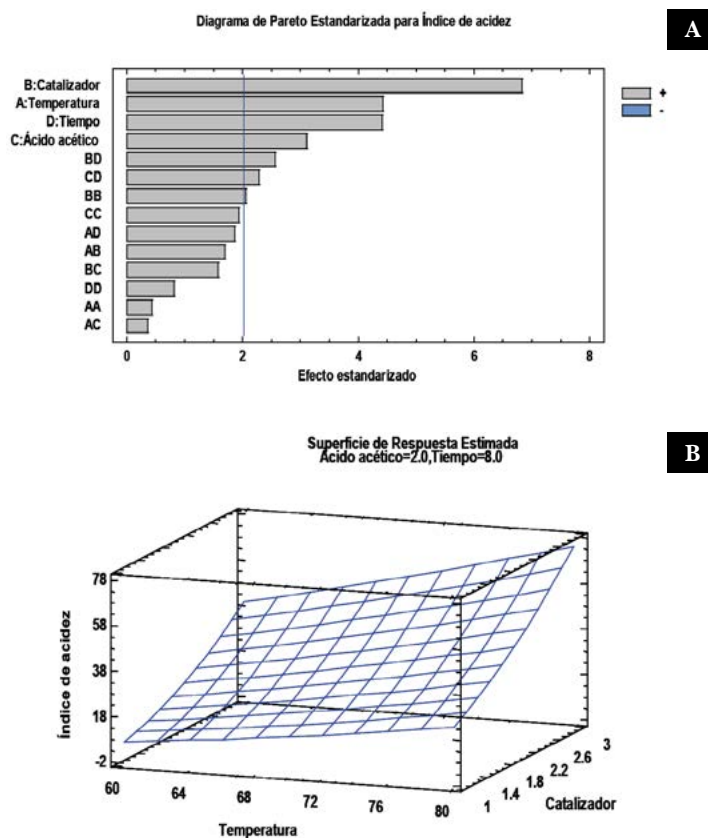


Figure 2. A) Pareto chart obtained from the statistical analysis of the acidity index values, where the effect of temperature, catalyst, acetic acid, and time can be observed. B) Response surface plot for the highest acidity index for the epoxidation and hydrolysis reaction.

$$\begin{aligned} \text{Acidity index} = & 337.045 - 3.40554 * \text{temperature} - 73.0987 * \text{catalyst} - 110.777 \\ & * \text{acetic acid} - 35.6272 * \text{time} + 0.0100476 * \text{temperature}^2 + 0.450605 * \text{temperature} \\ & * \text{catalyst} + 0.191283 * \text{temperature} * \text{acetic acid} + 0.248587 * \text{temperature} \\ & * \text{time} + 4.73505 * \text{catalyst}^2 + 8.40307 * \text{catalyst} * \text{acetic acid} + 3.41624 * \text{catalyst} \\ & * \text{time} + 17.8879 * \text{acetic acid}^2 + 6.07902 * \text{acetic acid} * \text{time} + 0.472183 * \text{time}^2 \end{aligned}$$

As mentioned earlier, the acidity index of chicken fat was 0.89 mg KOH/g, and after the epoxidation and oxirane ring opening reaction, the product increased to 40.63 mg KOH/g, confirming the presence of hydroxyl groups corresponding to the polyol. The FTIR spectrum of the synthesized polyol shows the band at 3432.30 cm^{-1} of OH groups, at 2922.63 cm^{-1} and 2853.90 cm^{-1} of symmetrical stretching C–H, at 1739.90 cm^{-1} of ester carbonyl, at 1460.27 cm^{-1} of asymmetric bending C–H, and at 1097.56 cm^{-1} and 1159.31 cm^{-1} of symmetrical and asymmetrical stretching of C–O–C. The observed bands are similar to the spectrum of the polyol by Ionescu *et al.* (2011), who synthesized polyester polyols from castor oil. In the FTIR spectra at variable temperature (Figure 3A), variable catalyst concentration (Figure 3B), and variable reaction time (Figure 4), it is observed that under low reaction conditions ($60 \text{ }^\circ\text{C}$, 1% catalyst, 4h), the signal of epoxy groups at 827 cm^{-1} , corresponding to the C–O–C stretching of the oxirane ring, is observed, as well as the absence of signals of hydroxyl groups, confirming the generation of triglyceride epoxides. According to Hazmi *et al.* (2013), the reaction temperature has

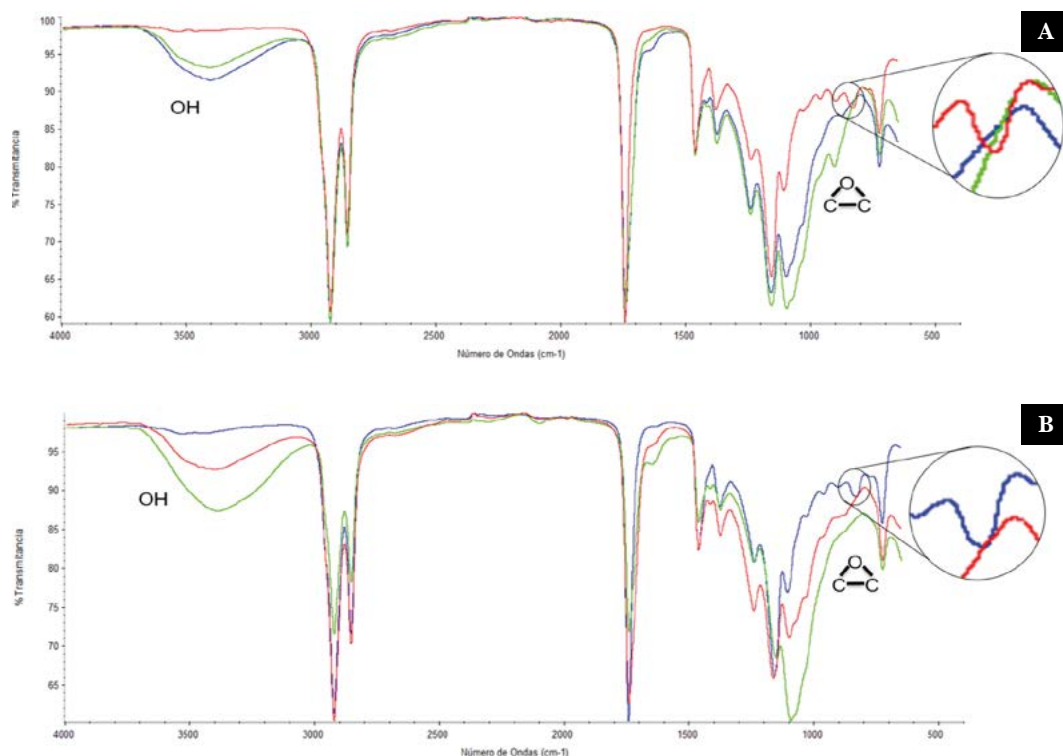


Figure 3. A) FTIR spectrum of the epoxidation reaction and polyol synthesis. Reaction temperature at $60 \text{ }^\circ\text{C}$ (red), $70 \text{ }^\circ\text{C}$ (green), and $80 \text{ }^\circ\text{C}$ (blue). B) FTIR spectrum of the epoxidation reaction and polyol synthesis. Catalyst at 1 mol (blue), 2 mol (red), and 3 mol (green).

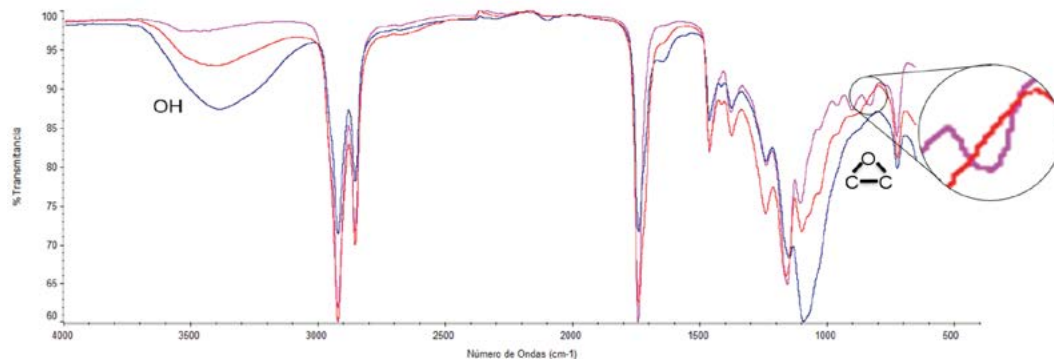


Figure 4. FTIR of the epoxidation reaction and polyol formation. Reaction time: 4 hours (pink), 6 hours (red), and 8 hours (blue).

an impact on the formation of the epoxide group because increasing the temperature can generate ring-opening byproducts.

In the most severe reaction conditions (80 °C, 3% catalyst, and 8h), the signal corresponding to the epoxide group disappears, and there is a higher intensity of signals from hydroxyl groups between 3400-3600 cm^{-1} , indicating the cleavage of the oxirane ring (Milchert *et al.*, 2016).

Thi *et al.* (2015) epoxidized palm oil to synthesize polyols at different reaction times and the same temperature in the presence of water. They observed that at shorter reaction times, there was no oligomerization of the epoxy groups (844 cm^{-1}). However, at longer reaction times, this signal decreased and disappeared at 6 hours, with new stretching bands observed at 3240-3670 cm^{-1} corresponding to the hydroxyl groups. Purwanto *et al.* (2010) reported that the oxirane ring can be hydrolyzed due to the reaction mixture containing H_2SO_4 , HAc, and water, which are rich sources of protons that react with the oxygen atoms of the oxirane rings. This can be confirmed by the ^1H NMR spectrum of the polyol obtained from the epoxidation and hydrolysis reaction (Figure 5), which shows a signal assigned to the $-\text{C}=\text{C}-\text{H}$ proton group between 5 and 6 ppm.

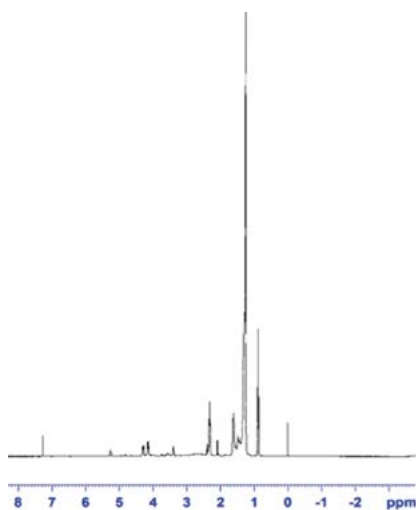


Figure 5. ^1H NMR spectrum of the polyol obtained from chicken fat.

Saalah *et al.* (2017) indicate that oligomerization is a side reaction of epoxide ring opening, which occurs when the epoxy group tends to react with acid or water, followed by the dimerization of the already formed hydroxyl compound, where the first step in the reaction is the attack of a proton on the epoxy group, which leads to the formation of hydroxyl and acetyl or formyl groups.

CONCLUSIONS

Chicken fat is a viable source for the formation of polyols through the epoxidation reaction followed by oxirane ring opening. This is feasible due to the fatty acid profile of chicken fat, which confirms the majority presence of palmitic, oleic, and linoleic acids, useful for polyol formation. The optimization of the epoxidation reaction followed by oxirane opening was favorable, demonstrating that all factors influence polyol formation. The higher concentration of catalyst (3 mol), acetic acid (2 mol), temperature (80 °C), and reaction time (8 h) favor the formation of polyols from chicken fat as the formed epoxides are completely hydrolyzed.

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