

Evaluation of the polyol/diisocyanate molar ratio in the physicochemical properties of biopolyurethane produced from poultry fat

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— Abstract —

Conventional polyurethanes are the most demanded plastics in the world, they are manufactured from petroleum derivatives which is a non-renewable resource and contributes to environmental pollution. However, biopolyurethanes are produced from renewable sources, such as vegetable oils or plant-derived sugars, which help reduce dependence on fossil fuels and decrease the carbon footprint. The synthesis of biopolyurethanes makes it possible to adjust the properties of the final materials to suit different applications such as the automotive industry, construction, furniture manufacturing, and textile industry. They can vary in terms of strength, elasticity, stiffness, and load capacity. These characteristics depend on the chemical structure and concentration of the polyol and diisocyanate with which they are produced.

This paper describes the synthesis of polyurethanes using polyol from poultry fat and isophorone diisocyanate and the effect of the polyol/isocyanate (OH/NCO) ratio on the physicochemical properties of polyurethanes obtained. In the infrared spectra (FTIR) was observed the presence of the band 3350 cm^{-1} corresponding to the -NH bond and the absence of stretching of the isocyanate group (NCO) to 2270 cm^{-1} , indicating that all isocyanate groups reacted during polymerization. Polyurethanes generated with molar ratios polyol/isocyanate (OH/NCO) 1: 3, 1: 4, and 1: 5 were evaluated, observing that the higher the molar ratio increases the rigidity and compressive strength of biopolyurethane, probably due to the greater formation of crosslinks.

Keywords:

Bioplastics; waste; stiffness; compression.

Polyurethanes are versatile polymeric materials that, from the synthesis developed by Bayer et al. in 1937, have been used to generate products with different characteristics such as resins, coatings, flexible and rigid foams, elastomers, and adhesives. Among these applications, rigid and flexible foams have been mainly used in the transportation, construction, packaging, and furniture industries. Polyurethanes are polymers that are generally produced by polyaddition reactions between polyols and isocyanates, which, depending on their structures, will be the physical-mechanical properties of the polyurethane obtained (Shen, et al. 2019). For example, Kasprzyk and Datta (2019) observed that the concentration of isocyanate groups in the prepolymer affects the degree of phase separation of the hard segment that can be correlated with the chemical structure and mechanical-dynamic properties of the polyurethane. Moreover, Zieglowski et al. (2019) researched the reactivity of Kraft lignin modified with different commercial isocyanates in the reaction with conventional polyols, identifying that the morphology, modulus of elasticity, and density are different in all the polyurethanes obtained. Daneshvar et al. (2019) prepared adhesive polyurethanes using poly 4,4'-diphenyl methane diisocyanate and toluene diisocyanate (TDI) in different NCO/OH and sawdust polyol ratios, observing that by increasing the NCO/OH ratio to 1.7, the shear force increased.

As can be seen, different efforts have been made to develop biopolyurethanes using polyols or isocyanates of biological origin. The synthesis of polyols from vegetable oils such as sunflower, fig, soybean, castor, palm, olive, flaxseed, grape seed, corn, jatropha, and rice bran oil are mainly described (Ang et al., 2014; Calvo-Correas et al., 2015; Fu et al., 2019; Ismail et al., 2011; Narine et al., 2007; Purwanto, 2010; Rosnah et al., 2016; Shen et al., 2019; Valero et al., 2008). However, these oils, since they are edible, generate controversy regarding their use for industrial purposes. Poultry fat has been used as a raw material for the production of biodiesel, biolubricants, and polyols (Galeano & Guapacha, 2011; Rojas & Girón, 2011, Ramírez et al., 2023). This project proposes the use of polyol of poultry origin for the production of biopolyurethanes and its study on the effect of the OH/NCO molar ratio on their physicochemical properties.

METHODOLOGY

Synthesis of polyol from poultry fat

Poultry fat was extracted, purified, and characterized as described by Hernández-Cruz et al. (2015). The methodology described by Ramírez, et al. (2023) was used to obtain the polyol. wherein the poultry fat was epoxidized by adding acetic acid and hydrogen peroxide in stoichiometric relation

to the number of moles of double bonds present in the oil. To determine the number of moles of the double bonds of poultry fat, the iodine value was used according to the following formula (equation 1):

$$(1) \quad mol(=) = IY / (2 \times Mi \times 100) \times P$$

Where:

mol (=) mol of the double bonds present in the oil

IY = $63.09 I_2 / 100g$.

Mi = molecular weight of iodine in grams.

P = weight of sample in grams.

Subsequently, according to the calculations obtained, the sample was placed in a 3-neck flask, acetic acid (CH_3COOH) was added, and as a catalyst H_2SO_4 . The reaction was kept under constant stirring (350 rpm) in an oil bath at a temperature below $30^\circ C$, then at this same temperature H_2O_2 was added dropwise over 30 minutes; once added, the temperature was increased and maintained for 6 h. After the reaction time, 5% v/v sodium bicarbonate was added, stirred and the contents of the flask were placed in a separatory funnel to perform washes by adding 10% $NaHCO_3$ twice, then water, and finally 5% $NaHCO_3$ until neutrality was reached, which was measured with a model potentiometer Session 3 (HACH, Colorado, USA) with a model pH electrode 51935-00 (HACH, Colorado, USA). Finally, 20% w/w anhydrous Na_2SO_4 was added to the organic phase to remove any remaining traces of water from the washes (Salimon et al., 2014).

To characterize the synthesized polyol, an IR spectroscopy was performed using an IR Nicolet™ spectrophotometer (Thermo Scientific, Massachusetts, USA) in the range of 650-4000 cm^{-1} with a resolution of 4 cm^{-1} and 16 scans (Jayavani et al., 2017). The structure of the polyol was confirmed by ^{13}C nuclear magnetic resonance (NMR) using a model DD2 (Agilent, California, USA) of 500 MHz. Experiments were performed at $25^\circ C$ and deuterated chloroform as solvent. After dissolving 5 mg of the sample, approximately 1 mL of the solution was transferred to a 5 mm NMR tube. The sample tube was inserted into the magnet and allowed to reach thermal equilibrium for 10 minutes before the experiment.

The acid number was performed according to the official AOCs Te 1a-64 method (2009). For the determination of the hydroxyl number of the polyol, the methodology described by Zhang et al., (2015) was used. 1 g of sample was placed in a 50 mL flask, 10 mL of tetrahydrofuran (THF) was added, and the contents were mixed with the aid of a magnetic stirrer; to this solution, 10 mL of catalyst solution (1 g of pyridine in 100 mL THF) was added followed by 5 mL of acetylating solution (5 mL of acetic anhydrous in 50

mL THF) and stirred for 10 min at 25°C, then 10 mL of hydrolysis solution (20 mL of water in 80 mL THF) was added and the stirring continued for 30 min. This solution was titrated potentiometrically with the standard 1 N ethanolic KOH solution, using the potentiometer model Session 3 (HACH, Colorado, USA), with a pH electrode model 51935-00 (HACH, Colorado, USA) until the first pH change was observed. Calculation of the hydroxyl number was obtained by equation 2:

$$(2) \quad OHN = \frac{(V_2 - V_1 * N * 56.11)}{m} + AN$$

Where:

OHN is the hydroxyl number value

V_2 is the volume of KOH spent during blank titration

V_1 is the volume of KOH spent during sample titration

N is the normality of the KOH standard solution

m is the weight of the sample, g.

AN is the acid value of the sample.

Obtaining polyurethanes

Polyurethanes (PUs) were obtained according to the methodology described by Zhang et al., (2014) with modifications. The synthesized polyol was mixed with isophorone diisocyanate (IPDI) in OH:NCO molar ratios of 1:3, 1:4, and 1:5, using ethyl methyl ketone (EMK) as solvent (5mL). The components were brought to 70°C and mixed continuously for 30 min. Subsequently, the mixture was poured into 8 cm x 4.5 cm (length x width) silicone molds, which were dried for about 14 days. Finally, the PU sheets were cut into specific dimensions for mechanical testing.

The polyurethanes obtained were characterized by Fourier transform infrared spectroscopy according to the methodology described above.

Compression properties were evaluated according to the methodology described by Acuña et al. (2021), using an EZ-SX model texturometer (Shimadzu, Maryland, USA) following the ASTM D1621 standard with a sample size of 5 cm x 5 cm x 3 cm and a speed of 3 mm/min.

RESULTS AND DISCUSSION

The polyol synthesized under the conditions described had an acid value of 12.63 mg KOH/g (Table 1), a value higher than that observed in the poultry fat used as raw material. This increase in acid number checks for the presence of acidic hydrogens, i.e. hydroxyl groups (OH) in the reaction product corresponding to the polyol.

On the other hand, the quantification of the hydroxyl number (OHN) for the polyol was 70 mg KOH/g, a value close to the prediction of the model described by Ramírez (2023), which was 78 mg KOH/g. Comparing the hydroxyl number values with other investigations that produce polyols from vegetable fats, the hydroxyl number values obtained in this study are lower (Table 1).

Table 1

Acid index and hydroxyl number of the polyol from chicken fat and polyols from vegetable oils (Shen et al. 2019).

Sample	Acidity index (mg KOH/g)	Hydroxyl number (mg KOH/g)
Raw matter (chicken fat)	0.89	-
Chicken Fat Polyol	12.63	70
Soybean Oil Polyol	-	150.4
Flaxseed Oil Polyol	-	190.8
Olive Oil Polyol	-	190
Grapeseed Oil Polyol	-	270
Rice Bran Oil Polyol	-	232

This difference in results may be due to three factors; firstly because the vegetable oils used for this purpose have higher percentages of polyunsaturated fatty acids in their triglycerides (Table 2); therefore, there is a greater likelihood that these double bonds generate a greater number of hydroxyl groups (Marcano Serrano, 2008). A second factor is that, in the reaction, some hydroxyl groups formed in the triglyceride were nucleophilically added to a nearby epoxide group, forming an ether group. On the other hand, the side reactions associated with ring opening caused by the components present in the reaction mixture such as acetic acid, protons, acetic peracid, or hydrogen peroxide, generate non-hydroxylated compounds or with fewer hydroxyl groups, which would generate less quantification (Rangarajan, 1995).

Table 2

Percentage of polyunsaturated fatty acids from chicken fat and different vegetable oils (Marcano Serrano, 2008)

Sample	Linoleic acid (%)	Linoleic acid (%)
Chicken fat (this study)	17.834	1.180
Soybean oil	53	8
Linseed Oil	16	52
Olive oil	6.3	0.3
Palm Oil	52.1	7
Rapeseed oil	14.5	11

To study the structure of the polyol, functional groups present were identified by ATR-FTIR (Figure 1). The bands observed in the polyol of this work are similar to those observed by Ionescu et al. (2011), who prepared polyester polyols by esterification from castor oil and petrochemical polyesters (adipic acid and diethylene glycol).

The band at 3432.30 cm^{-1} is assigned to hydroxyl groups, while the bands at 2922.63 cm^{-1} and 2853.90 cm^{-1} are assigned to symmetrical stretching in the C-H phase. The sharp intense band at 1739.90 cm^{-1} is characteristic of the ester carbonyl, the band at 1460.27 cm^{-1} is assigned to asymmetric C-H bending, and the bands at 1097.56 cm^{-1} and 1159.31 cm^{-1} to symmetric and asymmetric C-O-C stretching.

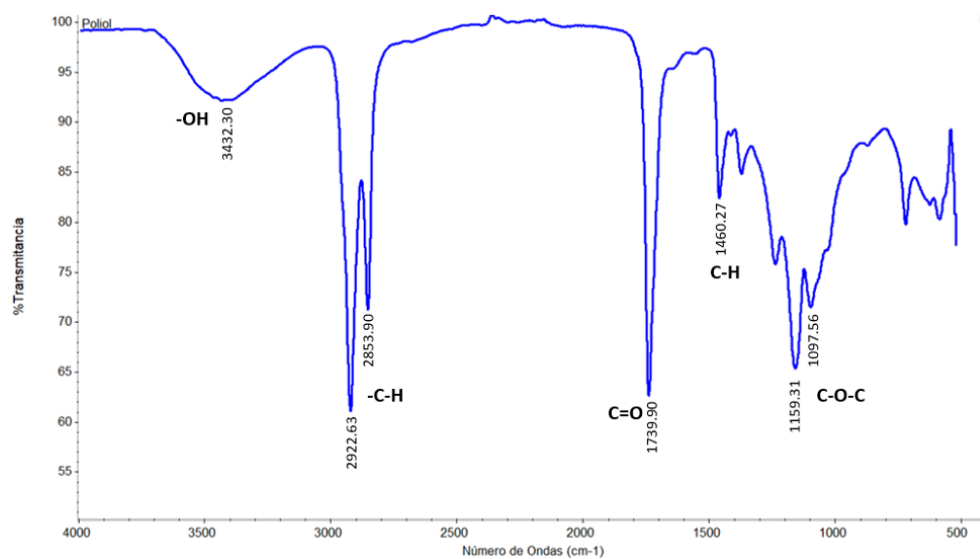


Figure 1. Polyol FTIR spectrum from chicken fat

On the other hand, when evaluating the FTIR spectra (Figure 2) of the synthesized polyurethanes, it is observed that (OH/NCO molar ratio 1:3, 1:4, and 1:5) the 2270 cm^{-1} signal band is not present, associated with the stretching vibration of the $\text{N}=\text{C}=\text{O}$ group of the IPDI, indicating that all the isocyanate groups reacted during the polymerization with the polyols. Also observed in the PU spectra is a band centered at 3350.63 cm^{-1} , 3351.95 cm^{-1} , and 3341.13 cm^{-1} respectively, which was attributed to N-H stretching of the urethane group, this becomes more intense as the isocyanate content in the material increases, due to increased formation of urethane groups. In addition, two bands could be observed in the region of amide II assigned to the stretching vibration of C-N together with the vibration in the plane of the N-H bond, present in the product of molar ratio 1:3 at 1644.74 cm^{-1} and 1552.68 cm^{-1} ; in that of molar ratio 1:4 at 1643.27 cm^{-1} and 1554.00 cm^{-1} and for the ratio 1:5 at 1640.79 cm^{-1} and 1552.89 cm^{-1} . This band also becomes more intense with the increase in isocyanate content due to the greater participation of urethane groups.

The bands could be seen at 1734 cm^{-1} attributed to the stretching of the carbonyl of the ester groups (C=O). These results indicate that the IPDI reacted completely with the polyol's hydroxyls and produced polyurethane (Chen et al., 2021; Dai et al., 2020; Mathew et al., 2017).

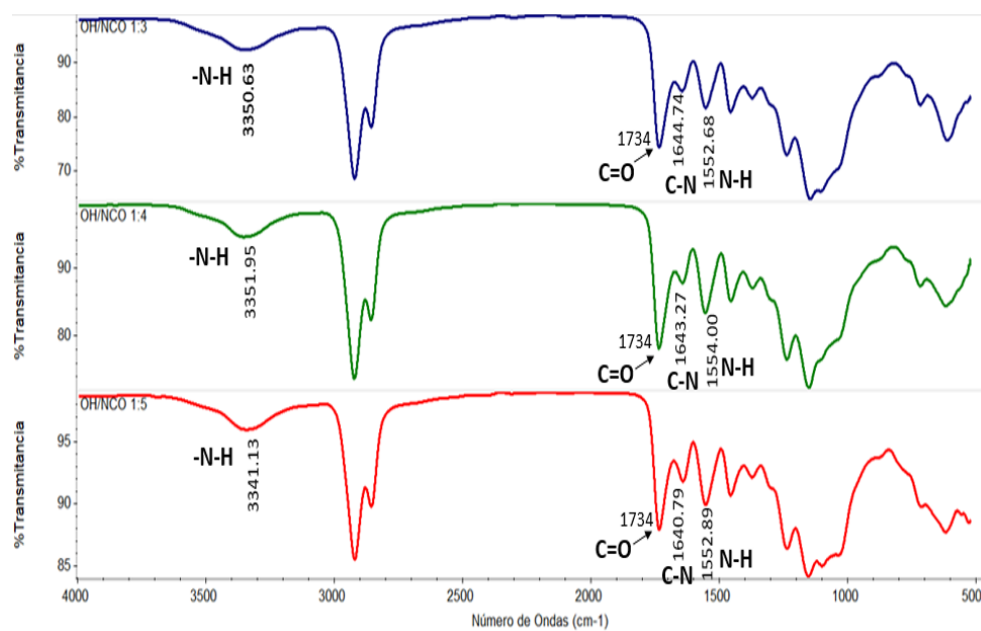


Figure 2. FTIR spectrum of the polyurethanes obtained at different molar ratios of the OH/NCO groups

The high reactivity of the isocyanate group with the active hydrogen compounds can be explained by the following resonance structures (Figure 3):

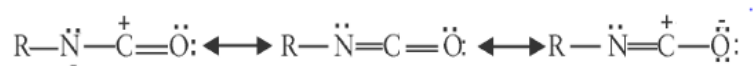


Figure 3. Canonical structures generated by the resonance effect of the isocyanate group

The electron density is highest on the oxygen atom, while the carbon atom has the lowest electron density, making the carbon atom positively charged and the oxygen or nitrogen negatively charged depending on the canonical structure.

The reaction of isocyanates with active hydrogen (OH) compounds is an addition to the carbon-nitrogen double bond (Figure 4). This reaction takes place as follows: the nucleophilic center of the active hydrogen compounds, i.e. the oxygen atom of the hydroxyl groups or the nitrogen atoms in the case of amines, nucleophilically attacks the electrophilic carbon atom, and hydrogen is added to the nitrogen atom of the -NCO groups. Electron-removing groups increase the reactivity of -NCO groups and, on the other hand, electron-donating groups decrease the reactivity towards active hydrogen compounds (Ionescu, 2016).

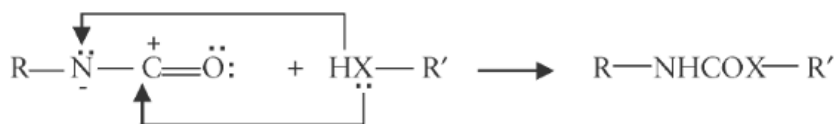


Figure 4. Reaction mechanism of the isocyanate group with active hydrogen compounds

To evaluate the effect of the OH/NCO molar ratio on the formulation of the polyurethanes, the mechanical compression test was carried out.

Of the three samples generated, the PU sample with a molar ratio of 1:3 (OH/NCO) did not meet the requirements for the compression method, because the material obtained was a material that hydrated easily, which made it unmanageable, preventing quantification, while the polyurethanes prepared from the 1:4 and 1:5 OH/NCO molar ratios were solid foams and therefore it was possible to perform the determination. Nguyen Dang et al (2016) mention that the higher the OH/NCO ratio, the greater the increase in hard segments, generating more crosslinking in PU polymerization.

Figures 5 and 6 show the graphs of the force exerted in Newton (N) and the displacement of the material (mm), a parameter described as stiffness, which is a quantitative measure of the opposition to elastic deformation due to force or stress. The material obtained with the ratio 1:4 (OH/NCO) had a displacement of 15.478 mm applying stress of 400.753 N, so the resulting stiffness was 25.89 N/mm, while for the polyurethane obtained with a ratio 1:5 (OH/NCO), applying the same stress, displaced 9.652 mm so the stiffness

was 41.52 N/mm. This indicates that at a lower 1:4 (OH/NCO) ratio, the stiffness was lower, compared to the PU with a 1:5 (OH/NCO) ratio. This result may be because, in PU 1:5, there is a greater amount of isocyanate groups that generate greater cross-linking with the polyol, which generates greater resistance to deformation compared to the PU obtained with the 1:4 ratio.

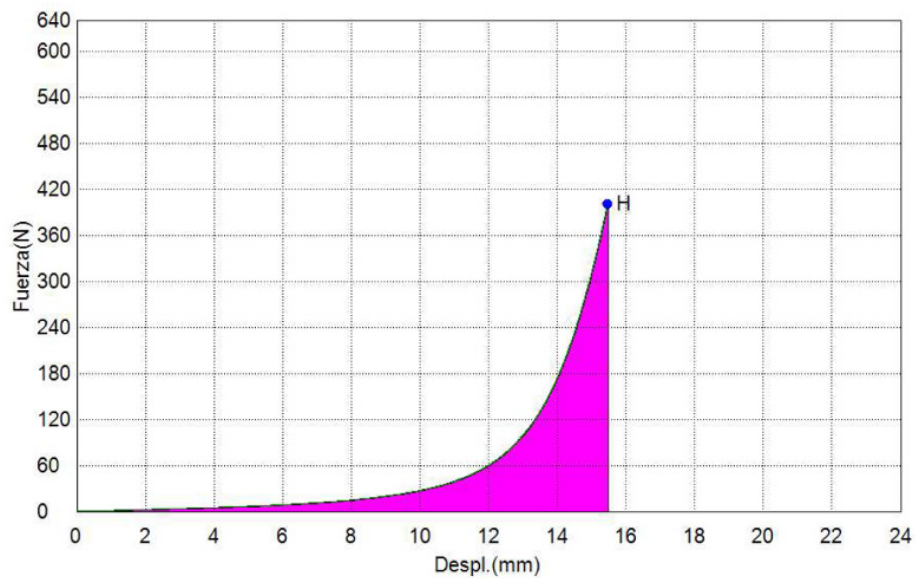


Figure 5. Stiffness of polyurethane generated with molar ratio 1:4 (OH/NCO)

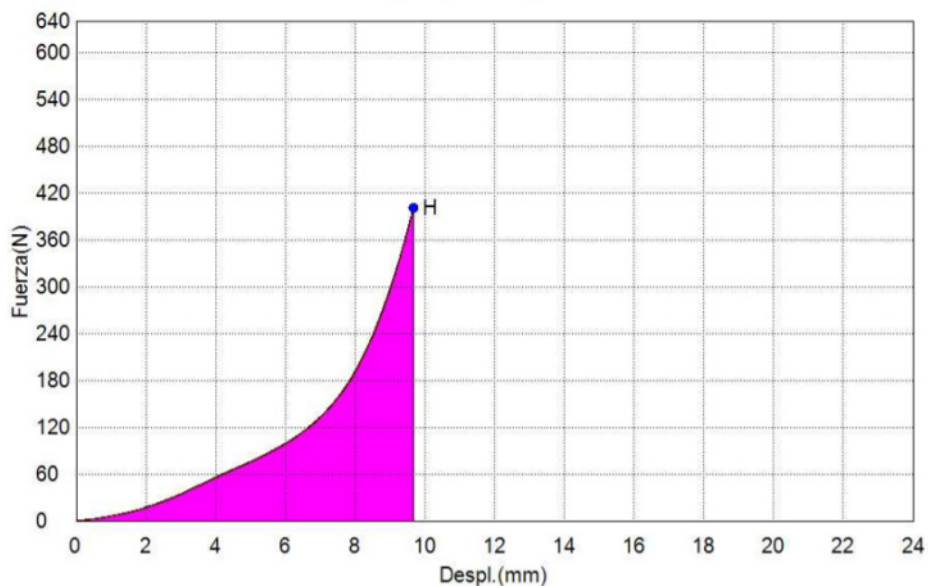


Figure 6. Stiffness of polyurethane generated with molar ratio 1:5 (OH/NCO)

This same behavior is observed in the quantification of the compressive strength, with a higher value being observed for the material generated with the ratio 1:5 (0.4152 ± 0.003 MPa) about that obtained with the ratio 1:4 (0.2589 ± 0.001 MPa).

The compressive strength of chicken fat polyurethanes was compared with the research of Acuña et al., (2020) (Table 3), who prepared rigid polyurethane foams based on castor oil with flame retardant properties and high insulation capacity. They prepared PU from castor oil polyol (BIO1), PU from castor oil with surfactants and foaming agents (BIO2), and PU from castor oil with surfactants and flame retardant properties (BIO2/EG).

BIO1 foam obtained low compressive strength because it had a porous structure and large cells, which favored the fragility of the cell walls. By modifying the polyol with a surfactant, the compressive strength increased because the viscosity increased by the interaction of the higher number of OH groups of the biobased polyol with the isocyanate groups, improving the network structure, generating more closed cells and increasing the crosslinking density.

When the flame retardant agent is added, the compressive strength decreases, causing poor interfacial adhesion, and damaging cell walls and foam integrity. For this reason, the compression values obtained in this work are higher than bio-based polyurethanes with additives, confirming the presence of a homogeneous mixture in the polymerization.

Table 3

Compressive strength of PU from chicken fat, castor oil, and fossil origin

Sample	Compressive strength (MPa)
PU1:4 from chicken fat (This work)	0.2589 ± 0.001
PU1:5 from chicken fat (This work)	0.4152 ± 0.003
BIO1 from castor oil (Acuña, 2020)	0.071
BIO2 from castor oil (Acuña, 2020)	0.146
BIO2/EG from castor oil (Acuña, 2020)	0.112
PU foam to petroleum base (Li et al., 2015)	0.137 – 0.310

Comparing the compressive strength values with those of polyurethane foams generated from petroleum, it is observed that PU 1:4 and those obtained with castor oil are within the range obtained for petroleum-based PU; while PU 1:5 is higher, demonstrating that it is possible to obtain polyurethane foams from chicken fat polyols with properties similar to those of petroleum-based foam.

The scaling up of the polyurethane production process from poultry fat reveals the search for sustainable and environmentally friendly materials.

However, the feasibility of this process for commercial exploitation depends on several key factors that must be carefully considered such as the quantity and quality of poultry fat, the investment for process optimization, the evaluation of the costs associated with production, the analysis of the market to compete in price and quality with conventional polyurethanes, and the evaluation of the environmental impact on their production.

CONCLUSIONS

The chicken fat polyols generated by epoxidation and *in situ* hydrolysis were appropriate for the formulation of biopolyurethanes since the number of hydroxyl numbers of the polyol and the molar ratio of NCO used favored the polymerization of the PU synthesis, therefore, the FTIR spectra indicated the formation of the PU, which indicated that the hydroxyl groups had reacted with the NCO group forming the hydrogen bonds.

The OH/NCO molar ratio is of utmost importance for the synthesis of PU, since its mechanical properties depend on it, the higher the hydroxyl value, the more flexible products will be generated, and the higher the NCO value, the less ductile and more rigid products will be generated.

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