



Synthesis of Flexible Polyurethane Foam Based on Palm Oil with Enhanced Characteristics

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Abstract

Conventional polyols are derived from petroleum derivatives. The limited availability of petroleum cannot keep up with the increasing human needs. Palm oil is a potential alternative to replace petroleum as a source of polyols. However, palm oil polyols have a weakness, namely having more secondary OH groups than primary OH groups, so their reactivity to isocyanates is low. This condition produces polyurethane foam collapse, rigid and brittle. Therefore, it is necessary to research to examine the level of comparison of palm oil polyols and commercial polyols in producing flexible polyurethane foam with the best characteristics. The treatments were the ratio of palm oil polyols and commercial polyols of 80%:20%, 70%:30%, 60%:40%, and 50%:50%. Parameters observed were functional group analysis using FTIR, visual observation, foam development, density, foam cell morphology observation using SEM, tensile strength, and elongation at break. The results showed that the ratio of 60% palm oil polyol and 40% commercial polyol resulted in foam with higher swelling, lower density, and better flexibility. This research has produced flexible polyurethane foam from palm oil polyol with a more than 50% composition.

Keywords: Biopolyol; collapse; elongation at break; tensile strength

1. Introduction

Polyurethane is formed from the reaction of polyether or polyester, which has an OH group, and isocyanate, which has an NCO group, to create a urethane bond [1]. Various polyurethane products include coatings, foams, elastomers, and adhesives [2]. Polyurethane foam has the largest market share compared to other polyurethane products. There are three types of polyurethane foam: flexible, semi-rigid, and rigid. The type of polyurethane foam is determined by the composition of the hard and soft segments, which affects its mechanical properties. Physically hard segments have cross-links that provide rigidity to the foam, while soft segments provide elastic properties [3]. The demand for flexible

polyurethane foam is increasing along with the increase in the furniture, carpet, mattress, construction, packaging, and automotive interior industries because it has low density, elasticity, and strength [3,4,5].

Flexible polyurethane foam is a polymer synthesized from polyol (-OH), diisocyanate (-NCO), and a blowing agent [7]. The polyols used are derived from petroleum derivatives. The availability of petrochemical polyols is getting less because they are non-renewable. Developing renewable polyols is one alternative to ensuring the sustainability of polyols as polyurethane raw materials. Renewable polyols can be obtained from biomass found in nature, such as triglycerides (oils), carbohydrates, polyphenols

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(lignins and tannins), and organic wastes, such as crude glycerol [8]. Furthermore, [8] explained that the aromatic OH contained in the phenol group is 300-1000 times less reactive than primary and secondary aliphatic OH, so it is necessary to chemically modify phenol to be used as a source of polyol. Polyols containing aromatic OH can increase foam stiffness [9]. Likewise, carbohydrates must be modified first into polyols because they are solid at room temperature. Crude glycerol has a hydroxyl number higher than 100 mg KOH/g, which is 350 mg KOH/g which is not by the requirements of a polyol as a raw material for flexible polyurethane foam.

Palm oil is a potential source of polyols to be developed with simpler and cheaper conversion methods. The advantages of using palm oil as a source of polyols are its abundant availability, low price, and high enough unsaturated fatty acids. The conversion of palm oil into biopolyol with a simple technology through the oxidation of unsaturated fatty acids into epoxidized oil and then the epoxide ring opening (oxirane ring opening) is performed using a ring-opening compound. The palm oil polyol reaction was controlled to produce a biopolyol with a hydroxyl number of less than 100 mg KOH/g which is a requirement to produce flexible polyurethane foam [10].

Preliminary research results show that 100% palm oil polyol produces polyurethane foam that is collapsed, brittle, does not expand, and is not flexible. Palm oil polyols with secondary OH groups cause biopolyols to be less reactive to isocyanates, so compounds are needed that can increase the reactivity of the polyol system by mixing biopolyols with commercial polyols. [11] used a ratio of palm oil polyols and commercial polyols of 25%:75% to produce flexible polyurethane foam. [12] used a polyol ratio of olive oil and commercial polyol of 35%:65% to produce semi-flexible polyurethane foam. The studies still used commercial polyols of more than 50%, because the biopolyols used had a hydroxyl number of more than 100 mg KOH/g. The advantage of this research was using more than 50% biopolyol (palm oil polyol) with a hydroxyl number of less than 100 mg KOH/g. The mechanical properties of flexible polyurethane foam based on palm oil polyols are improved by mixing it with less than 50% commercial polyol. The mechanical properties of the foam are related to the density, expansion, and cellular structure of the foam. Therefore, it is necessary to

research to examine the level of comparison of palm oil polyols and commercial polyols in producing flexible polyurethane foam with the best characteristics.

2. Materials and Methods

Materials

Palm oil polyol synthesized at the Agroindustrial Processing Technology and Engineering Laboratory, Andalas University, commercial polyol (Sarfatika), DABCO BLV catalyst (Shandong Tonglan Chemical Co., Ltd.), DABCO T-9 catalyst (Tianjin Hutong Global Trade Co., Ltd.), silicone oil (Hubei Star Chem Co., Ltd.), TDI 80 (Shandong Baovi Energy Technology Co., Ltd.), distilled water, methylene chloride (Dow), polyethylene glycol 400 (Subur Kimia Jaya).

Palm oil polyol polyurethane foam synthesis – commercial polyol

The palm oil polyol used had a hydroxyl number of 99.37 mg KOH/g. The foam synthesis was carried out by mixing palm oil polyols: commercial polyols (80%:20%; 70%:30%; 60%:40%; and 50%:50%. The DABCO T-9 catalyst was added as much as 0.2 pphp, 0.53 pphp DABCO BLV catalyst, 1 pphp aquades, 4.2 pphp methylene chloride, and 1 pphp NIAX L-618 silicon oil). After that, the polyol system reacted with TDI-80 as much as 51.9 pphp. The mixture was stirred at 1200 rpm for 10 seconds. After that, it is poured into a mold of 120 x 120 x 100 mm³. After forming the foam, it is put into the oven at 70°C for 1 hour. Furthermore, the polyurethane foam is placed in a dry place at room temperature for 24 hours. Polyurethane foam products can be tested and analyzed (visual observation, density, expansion, and Fourier Transform Infra-Red analysis).

Foam Density (modification of [13])

The foam sample was cut with 2.5 x 2.5 x 1 cm³ and then weighed with an analytical balance 3 times. The data obtained were averaged.

Foam Development

Observations of foam development were carried out 3 times using the formula:

$$(\text{final volume}/\text{initial volume}) \times 100\%$$

Fourier Transform Infra-Red (FTIR)

Functional group analysis of polyurethane foam was carried out using Perkin Elmer FT-IR

Spectrometer Frontier L128-0099. The test sample is inserted into the holder and then scanned to produce a graph.

Mechanical Properties

Samples were prepared and measured with the HT-2402 Computer UTM testing machine, Cap.50KN, with a 5 mm/sec tensile speed. The test was carried out 3 times.

3. Results and Discussion

Different concentrations of commercial polyols added to the polyol system produce polyurethane foams with different characteristics. The characteristics of polyurethane foam are known from chemical analysis data, visual observations, physical tests, and mechanical tests.

Fourier Transform Infrared (FTIR)

FTIR analysis was used to determine the success of the polyurethane foam synthesis reaction by identifying the presence of functional groups in the polyurethane system. The FTIR spectrum of flexible polyurethane foam from the comparison of palm oil polyols and commercial polyols along with commercial flexible polyurethane foam as a control can be seen in Figure 1.

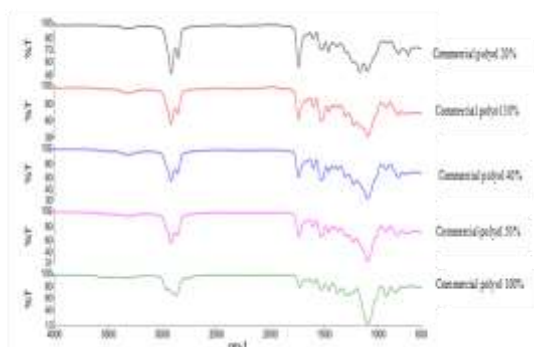


Figure 1. FTIR spectrum of polyurethane foam

Polyurethane foam from all treatments (20%, 30%, 40%, 50%, and 100% commercial polyol) had a stretching frequency of 3332 cm^{-1} - 3339 cm^{-1} , indicating the presence of -NH. Wave numbers 2921 cm^{-1} - 2969 cm^{-1} and 2847 cm^{-1} - 2872 cm^{-1} indicate C-H stretching vibrations and asymmetric and symmetrical stretching of the methylene group [14]. The absorption peak of -NCO at wave number 2267 cm^{-1} was not found in all treatments [15]. This shows

that the -NCO of TDI has reacted completely with the O-H group of the polyol [16]. The peak at wave number 1726 cm^{-1} - 1740 cm^{-1} indicates the presence of C=O groups in the urethane bond. Polyurethane foam treated with 20%, 30%, 40%, 50%, and 100% commercial polyol had an absorption peak at wave number 1517 cm^{-1} - 1529 cm^{-1} , indicating bending vibration of the -NH group [17]. Wave numbers 1170 cm^{-1} - 1293 cm^{-1} and 1095 cm^{-1} - 1102 cm^{-1} have C-N and C-O-C stretching vibrations (ether bonds from commercial polyols) [14,15]. The absorption peak at wave number 718 cm^{-1} - 756 cm^{-1} indicates the presence of HCN. HCN and -NCO (isocyanate) are toxic gases polyurethane foam releases when burned [16,5].

According to [5], in the synthesis of polyurethane foam using water as a blowing agent, there are 2 reactions, namely the reaction formation of urethane bonds (NCO & OH) and the reaction of formation of urea bonds (NCO and NH_2 as carbamic acid derivatives). The functional group NH_2 is obtained from the breakdown of carbamic acid into amine molecules and CO_2 . The peak of the C=O (urethane) functional group appears in wave number 1726 cm^{-1} - 1740 cm^{-1} , while the peak of the NH_2 functional group (urea) appears in wave number 1600 cm^{-1} - 1601 cm^{-1} [13]. The urea bond formation reaction is faster than the urethane bond formation reaction.

Visual observation

The flexible polyurethane foam produced by mixing palm oil polyols with commercial polyols (petrochemical polyols) can be seen in Table 1.

Table 1. Visual observations of flexible polyurethane foam

Palm oil polyol : commercial polyol	Observation
80%:20%	collapse, rigid, brittle, not soft, hollow
70%:30%	collapse, rigid, slightly soft, hollow
60%:40%	Slight collapse, flexible, soft, hollow
50%:50%	collapse, slightly rigid, slightly soft, hollow

Table 1 shows that the ratio of palm oil polyol and commercial polyol of 80%:20% and 70%:30% produces rigid polyurethane foam. In contrast, the palm oil polyol and commercial polyol ratio of 60%:40% produce flexible foam. Palm oil polyols have many secondary OH groups that are less reactive to isocyanates, so the urethane bond formation reaction takes a long time. Meanwhile, the formation of CO₂ from the reaction of water, methylene chloride, and isocyanate takes place quickly so that much CO₂ that is formed is released from the urethane system. This condition causes the foam to collapse and produce a rigid foam structure [21]. Palm oil polyols are polyesters that tend to produce rigid foams, while polyether polyols will produce flexible foams due to flexible ether bonds [8].

The commercial polyols by 40% can increase the reactivity of the polyol system to isocyanates so that the reaction speed of urethane formation can balance the rate of CO₂ formation reaction. The formed foam cells can trap most CO₂, creating open cells and producing a more flexible foam. The polyurethane foam produced from all treatments produces a hollow foam. Foam cavities or pores have 2 types of cells: open and closed. Rigid polyurethane foam has more closed cells and does not have interconnected pores, while flexible polyurethane foam has pores in the form of open cells and is interconnected [22].

Commercial polyol 50% in a polyol system produces a slightly rigid foam. The urethane formation reaction is somewhat faster, so less CO₂ is trapped in the foam cell. The foam cell traps a limited amount of CO₂ due to the rigidity of the cell wall, so the expansion of the foam cell volume is also limited.

Foam development

Foam development is closely related to the balance between blowing and gelling reactions [8]. The rapid development process can break down the thin cell walls resulting in open cells.

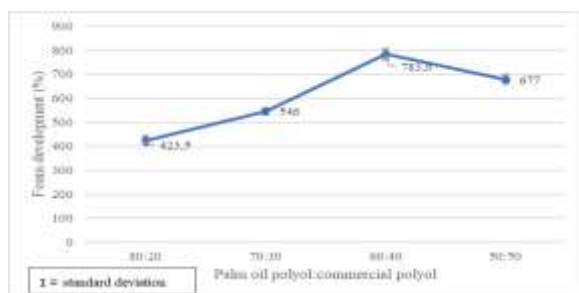


Figure 2. Development of polyurethane foam

Figure 2 shows that using 20% and 30% commercial polyols resulted in a less swelled polyurethane foam. This is because palm oil polyols have secondary OH groups that are less reactive to isocyanates, so the urethane (polymer) bond formation reaction becomes slow. The secondary hydroxyl group was 3 times slower to react than the primary hydroxyl group [23]. If the gelling reaction to synthesize the polymer is very slow compared to the CO₂ formation reaction, the foam cells will break, and the foam will fall back (collapse) before the polymer hardens [24].

The commercial polyols (petrochemical polyols) add up to 40%, increasing the polyol system's reactivity to isocyanates to balance the reaction speed of CO₂ formation. The use of commercial polyols of 40% resulted in maximum foam development because the polymer system could trap CO₂ as much as possible, resulting in higher foam development than other treatments. CO₂ comes from the reaction of water with isocyanates to produce carbamic acid, which then decomposes into CO₂ and amines [8]. This study also uses methylene chloride as a blowing agent because using 100% water as a blowing agent produces foam cells that break easily so that the resulting foam does not expand. The reaction of isocyanate-polyol and isocyanate-water produces heat, resulting in volatile methylene chloride forming more gas to develop the foam cell.

Commercial polyols 50% reduces foam development. This is because the gel formation in polymer synthesis is faster than the formation of CO₂, so less CO₂ is trapped in the stiff foam cells. However, the development of flexible polyurethane foam from palm oil polyols and commercial polyols was much lower than that of flexible polyurethane foam from 100% commercial polyols by 2784%. However, this study has succeeded in mainly using palm oil polyols (60%) as a source of polyols to replace petrochemical polyols.

Foam density

Density is an important parameter to indicate the foam type produced. The molecular weight and the hydroxyl number of the polyol used influence the density of the foam. The polyurethane foam densities of various grades of comparison of palm oil polyols and commercial polyols are shown in Figure 3.

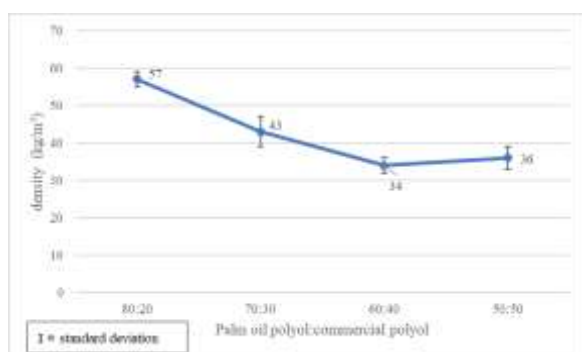


Figure 3. The density of polyurethane foam

The density of flexible polyurethane foam from 100% commercial polyols is 30 kg/m³. The density of flexible polyurethane foam is 34.4 kg/m³ [25]; 23 kg/m³ [20]; 23.5 kg/m³ [26]; 51.6 kg/m³ [27]; and 14 kg/m³ -55 kg/m³ [7]. The foam obtained from the ratio of 60% palm oil polyol and 40% commercial polyol produced foam with the lowest density (34 kg/m³). This density value belongs to the category of flexible foam.

Figure 3 shows that the higher the use of commercial polyols (up to 40%) in the polyol system, the lower the foam density. Comparison of palm oil polyols and commercial polyols were 80%:20%, 70%:30%, and 60%:40% produce foam with a density of 57 kg/m³, 43 kg/m³, and 34 kg/m³. Biopolyols have a lower molecular weight than commercial polyols. Low molecular weight biopolyols with higher hydroxyl numbers tend to produce higher-density foams [28]. Biopolyols have secondary OH groups, while commercial polyols classified as polyether polyols have primary OH groups. The primary OH group is more reactive than the secondary OH. The more primary OH groups in the polyol system, the more reactive it is with isocyanates to form soft segments, reducing the foam's cross-link density. Open cells containing more CO₂ are formed so that the foam expands more.

The commercial polyol 50% resulted in slightly increased foam density. The higher primary OH group content in the polyol system resulted in the gel formation reaction being faster than the CO₂ formation reaction, so the stiff foam cells became limited in trapping CO₂. This condition causes the cross-link density to increase.

Mechanical properties

The mechanical properties of the foam are related to its density. Foams with high tensile strength, yield

strength, and low flexibility show the mechanical properties of rigid foam and have a higher density [8].

Table 2. Tensile strength, yield strength, and elongation at break polyurethane foam

Palm oil polyol: commercial polyol	Tensile Strength (kPa)	Yield Strength (kPa)	Elongation at break (%)
80%:20%	280 ± 3.41	210 ± 2.68	29 ± 1.45
70%:30%	280 ± 1.82	100 ± 4.43	33 ± 3.51
60%:40%	200 ± 3.31	80 ± 1.52	57 ± 7.30
50%:50%	210 ± 2.43	110 ± 1.41	49 ± 6.24

Control foam (Commercial flexible polyurethane foam) has a tensile strength of 90 kPa, yield strength of 60 kPa, and elongation at break of 55%. Palm oil polyols and commercial polyols of 60%:40% produce foams with higher mechanical properties than commercial flexible polyurethane foams, namely tensile strength of 200 kPa, yield strength of 80 kPa, and elongation at break of 57%.

The ratio of commercial polyols up to 40% produces foams with lower tensile and yield strengths, increasing elongation at break (Table 2). This is because the palm oil polyol used has a hydroxyl number of 99 mg KOH/g which is higher than the hydroxyl number of commercial polyols. The hydroxy polyol number used to synthesize flexible polyurethane foam is 32 mg KOH/g – 37 mg KOH/g [13] or 15 mg KOH/g -100 mg KOH/g [8]. The polyol hydroxyl number greatly determines the type of polyurethane foam produced. The higher the hydroxyl number, the more isocyanate reacts to form rigid and semi-rigid foam. Hydrogen bonding between the segments in the urethane group is one factor affecting the mechanical properties [28]. Biopolyols have a lower molecular weight than petrochemical polyols. Lower molecular weight with higher hydroxyl number induces more reaction between isocyanate and OH group, forming more urethane and urea bonds. The increase in urethane and urea bonds forms cross-links in the foam, which is the leading cause of the increased hardness of the foam [29]. Thus, the more use of biopolyols or the less use of commercial polyols, the resulting foam with higher tensile strength and yield strength but lower foam flexibility. The use of biopolyol up to 40% produces foam with better flexible properties than other treatments. Commercial polyols can increase the polyol system's molecular weight in improving the foam's flexible properties.

The commercial polyols 20% and 30% produce polyurethane foam which is stiffer so that it has a

tensile strength (280 kPa; 280 kPa) and yield strength (210 kPa; 100 kPa). Rigid foam has stronger hydrogen bonds and requires greater force to break it.

The lower the hydroxyl number, the more flexible the polyurethane foam produced and the lower the tensile strength value. However, adding 50% commercial polyol caused the tensile strength and yield strength to be slightly higher, namely 210 kPa and 110 kPa, and the elongation at break decreased (49%). This is because the gel formation reaction (urethane) is faster than the formation of CO₂ so that only part of the CO₂ is trapped by the foam cells so that the cross-link density increases.

Scanning Electron Microscope (SEM)

The biopolyol component influences foam cell morphology through its reactivity, viscosity, nucleation capacity, surface tension, or the result of microphase separation [8]. Microphase separation in PUF generally explains defects in the foam, such as irregular pores or wrinkles. The morphology of polyurethane foam from various treatments was assessed using SEM. In Figure 4, it can be seen that all the foams have a pore structure with a combination of closed cells and open cells.

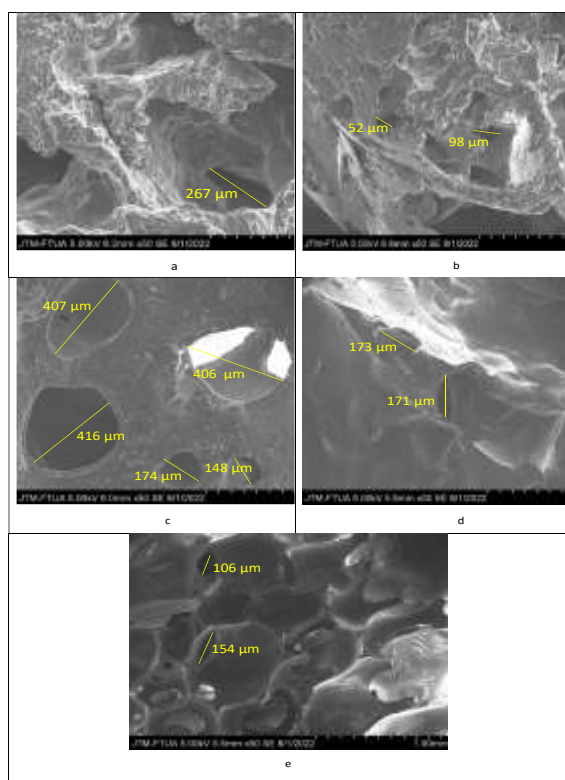


Figure 4. Morphology of flexible polyurethane foam from palm oil polyols and commercial polyols with a ratio of a)

80%:20%, b) 70%:30%, c) 60%:40%, d) 50%:50%, and e) 0%:100%

Figure 4 shows cell shapes are not round (a, b, and d). This indicates that during foam formation, collapse occurs. At the beginning of the foam synthesis reaction, expansion occurs due to the formation of CO₂ from the reaction of isocyanate, aquades, and methylene glycol. Then CO₂ is released from the cell, which causes the cell shape to shrink. Mixing palm oil polyol and commercial polyol up to 30% can reduce the size of the foam cells to 52 μm and 98 μm, but the number of closed cells is higher than the open cells, resulting in a rigid foam. The flexible polyurethane foam obtained from 60% palm oil polyol and 40% commercial polyol had a round cell shape. It tended to produce more open cells (Fig. 1c). Foam cell sizes are larger and tend to be homogeneous, namely 406 μm, 407 μm, 416 μm, 174 μm, and 148 μm. The size of this foam cell is almost the same as the results of the study of [27], which explained that the cell size of commercial flexible polyurethane foam is 476 μm. According to [5], the higher the development, the higher the flexible properties of the foam. However, in Figure 1d, adding 50% commercial polyol reduces the size of the foam cells to 171 μm and 173 μm, respectively. The size of the foam cells tends to be homogeneous, but the number of open cells is almost the same as that of closed cells. Figure 1e represents the open cell morphology of the foam derived from 100% commercial polyol. The foam cells look bloated. The cell sizes tend to be homogeneous. Namely, 106 μm and 154 μm, and the distance between the foam cells are very close. The cell size of flexible polyurethane foam from this study is almost the same as that of [30], which is 163.8 μm. Homogeneous cell size and more open cells are desirable characteristics of flexible polyurethane foams [27].

4. Conclusion

Flexible polyurethane foam has been successfully synthesized using the formulation of 60% palm oil biopolyol and 40% commercial polyol. The addition of commercial polyols has a significant impact on the properties of the final product. Commercial polyols increased the reactivity of polyol systems to isocyanates, enhancing the development and mechanical properties of flexible polyurethane foam. SEM showed more open cells and tended to be of homogeneous size. Flexible polyurethane foam based

on palm oil polyol has the potential to be applied to the automotive and furniture industries.

5. Conflicts of interest

“There are no conflicts to declare.”

6. Acknowledgments

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