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The Synergize effect of Chain extender to Phosporic acid catalyst to the ultimate property of Soy-Polyurethane

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The Synergize effect of Chain extender to Phosporic acid catalyst to the ultimate property of Soy-Polyurethane

Flora Elvistia Firdaus
Director of Research Jayabaya University
Dept. of Chem Eng, Jayabaya University-Jl Pulomas Kav 21 Jakarta 13201, Indonesia

E-mail: flora_elvistia@yahoo.com

Abstract. The polyurethanes (PUs) foam were made from vegetable oil; a soybean based polyol. The foams were categorized into flexible and semi rigid. This research is manufacturally designed polyurethane foams by a process requiring the reaction of mixture of 2,4- and 2,6-Toluene di Isocyanate isomers, soy polyol in the presence of other ingredients. The objective of this work was to functionalized soy-polyol using phosporic acid catalyst and chain extender, study their collaborative reaction in producing ultimate property of PU foam. Correlates the foam morphology images in accordance to mechanical properties of foams.

1. Introduction

Polyurethanes are an important group of polymers that submit chances to obtain the designed properties by proper selection of different composition [1]. The blends of polyol, isocyanate, and chain extender from low molecular gives rise to a multitude of forms which suitable for extremely different practical applications [2].

In the current years a fast growing interest in the development of bio-based products can reduce the widespread dependence on non renewable resources. Petroleum is non renewable, with inevitable depletion resources coupled with high cost has prompted researchers to develop alternatives to renewable based. Using vegetable oils and its derivatives for polyurethane are well known [3]-[4]. The nature of easy to degrade renewables and their derivatives, a symbol of environmental friendly has made them as an attractive candidates for development of sustainable technologies [5].

The properties of polyurethane (PU) foam can be modified within wide limits depends on raw material used. The major raw materials of polyurethane synthesis are polyols and isocyanates, which are largely influence to properties of foams and mainly contributes to bursting process of foam.The are requisite of the process that can decides the rigidity or flexibility of foams which are; chemical structure, equivalent weight, and functionality, and combination of polyols [6].

The preparation of polyols from vegetable oils for polyurethane and its application has been the subject of many areas of research. The very attractive of vegetable oils based polyols are its multiple hydroxyl functionality [7].

Vegetable oil-based polyols are traditionally prepared from triglyceride molecules. Polyols are successfully prepared using different methods. The most common methods are carbon-carbon double bonds which further oxirane ring-opening with alcohols or other nucleophiles [8]- [12].

Presenting phosporic acid as an appropriate catalyst in epoxy compounds, will react to reactive isocyanates forming oxazolidone during foaming process of polyurethane [13]. Most of rigid PU's are prepared from judicially formulated mixtures of polyol. The mixtures may contain a mixed polyols to the accomplishment ultimate properties. The benefit of using polyol mixtures for PU foam synthesis, it has minimum tendency to crystallize which further benefit to the better physical properties. The

difunctional such as 1,2 ethanediol or ethylene glycol are essential as chain extenders; in general is low molecular weight reactants produces hard segments; an increased intermolecular association or bonding effect [14].

The soft and hard part of multi-element array influenced the products obtained. The research was conducted to determine the synergize effects of phosporic acid catalyst in soy-epoxide hydroxylation reaction and chain extender to property of polyurethanes product. However, the reports did not explored the optimization amount of chain extenders; an ethylene glycol. Moreover, had studied the effect of blending of chain extender and polyol to properties of polyurethane. Chemical blowing agents and distilled water are extensively allow for the prior in PU foams synthesis, the responses to isocyanate has generated carbon dioxides and polyureas. The carbon dioxide causes of foaming which forming sectional structures. The practice of mixture of chain extender and polyol in making polyurethanes are to improve the properties and the foaming process which seldom studies.

The objective of the work is to determine the effect of phosporic acid to the effectiveness of chain extender in the synthesis to mechanical and the subtle changes of foams cellular images.

1. Experimental Procedures

2.1.Materials

The soybean oil was manufactured from local grocery. Hydrogen Peroxide and phosporic acid are obtained from Brataco Chemika, acetic acid from Merck Germany, and sulfuric acid from Tedia Company, Inc. All solvents and reagents were of laboratory grade and there were no treatments before used.

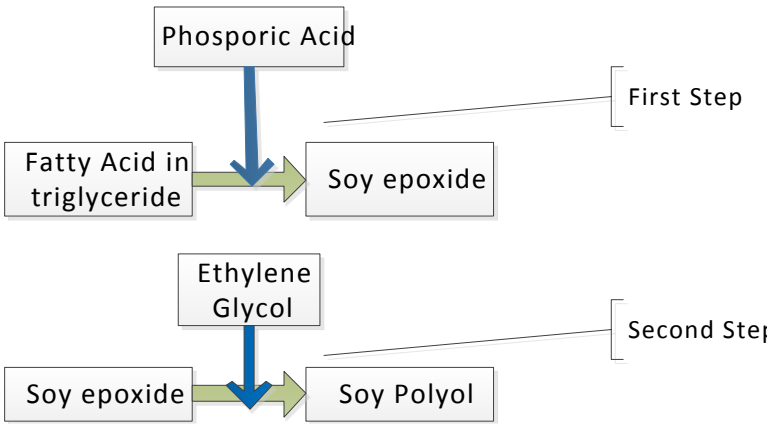

Figure 1. The Flowchart of the Process

For the first step, the unsaturated fatty acids in triglycerides chain of soybean oil are converted to soy-epoxide using phosphoric acid; having an oxirane rings. In the second step, the soy-epoxide with ethylene glycol are hydroxylated to soy-polyol.

Four blended of soy-polyols (SP); SP 1, SP 2, SP 3, and SP 4 are prepared from individual soy-polyol to achieve certain PU products, which then categorized. The catalyst were applied in the synthesis; phosporic acid with concentration 0,5% (v/v), 1,0% (v/v), and 1,5% (v/v) using three variables of temperature; 50^0C, 60^0C, and 70^0C. Another catalyst; sulfuric acid were used as for comparative. The soy-polyol were characterized before proceed to polyurethane synthesis, as can be seen in table 1.

Table 1. The Property of Material

Material	Properties	Value	Unit
Soybean Oil	1. Iodine Value	53,89	gr Iod/100 gr
	2. Viscosity	65,53	cps
	3. Acid Value	0,024	mgr KOH/ gr sample
Soy Polyol (SP)			
Temp 50 C			
SP 1	Oxirane Number	4,7	mgr KOH/ gr sample
SP 2		5,1	mgr KOH/ gr sample
SP 3		5,1	mgr KOH/ gr sample
SP 4		4,7	mgr KOH/ gr sample
Temp 60C			
SP 1	Oxirane Number	4,6	mgr KOH/ gr sample
SP 2		4,7	mgr KOH/ gr sample
SP 3		5,1	mgr KOH/ gr sample
SP 4		4,7	mgr KOH/ gr sample
Temp 70 C			
SP 1	Oxirane Number	4,7	mgr KOH/ gr sample
SP 2		4,7	mgr KOH/ gr sample
SP 3		5,1	mgr KOH/ gr sample
SP 4		4,7	mgr KOH/ gr sample

3.Methods

The property of polyol especially the physicochemicals have direct effect to the performances of polyurethane, as can be described below as important criterias for characterizing polyols.

3.1. Equivalent weight of Polyol

Equivalent weight more focused on polyols which has a weight distribution , the average equivalent weight can be calculated using the hydroxyl and acid number.

Equivalent weight of Blend $= \frac{56,1\ x\ 1000}{Hydroxyl number + Acid\ number}$ (1)

Acid number is calculated using = % weight of polyol A + % weight of polyol B (2)

So that

Equivalent weight of Blend $= \frac{100}{\frac{\%\ weight\ equivalent\ polyol\ A}{weight\ equivalent\ of\ polyol\ A} + \frac{\%\ weight\ equivalent\ B}{weight\ equivalent\ of\ polyol\ B}}$ (3)

3.2.Impact Resilience

The impact resilience of foam measures bouncing, elasticity, or springiness which describe % resilience or as % of return. The method has explained, percentage (%) of resilience of using two metal balls weighed 46 gr and 2,7 gr. The balls were dropped from 50 cm onto foam which every 5%

of return calibration is marked. Three drops were executed, and every three readings are averages counted.

3.3.The Obvious Density

The obvious density of foams are measured as per ASTMD 1622-03. The sample are prepared in scale $1cm^3$. The obvious density of of PU 1, PU 2, PU 3, and PU 4 were measured.

3.4.The Foam Morphology

The PU cellular images were studied by using Scanning electron microscope.

The synthesis of PU foam was done in a 400 mL glass reactor at normal pressure. The prepolymer product is a soy polyol was reacted to TDI isomers. It was designed for 2 hours of reaction. The result of the synthesized material was poured into a glass mold at room temperature for 26 hours.

3.5.Foam Preparation and Evaluation

The preparation of polyurethane foam were marked by its free rise method with formula which is shown in table 2.

Table 2. The formula of Polyurethane

Step 1 Epoxide: EG (mol/mol)	Step 2 Soy-polyol/H2O /TDI Isomers (70:30) (mol/mol)	Code of PU (Foam)
1:01	2,5:1:2,5	PU 1
1:05	2,5:1:2,5	PU 2
1:07	2,5:1:2,5	PU 3
1:09	2,5:1:2,5	PU 4

[a]The epoxide temp. synthesis are respectively 50,60, and 70^0C
[b]The elasticity of foams are influenced by polyol series
[c] Concentration of isocyanates are constant and stoichiometric.

4.Results and Discussion

4.1.The Effect of Chain Extender, catalyst, and temperature to Density

The synergize effect of phosporic acid catalyst to polyol synthesis using three different of temperatures; 50^0C, 60^0C, and 70^0C for PU synthesis, as can be seen in figure 2. The polyol was synthesized using four different ratios of epoxide to ethylene glycol; (1:1), (1:5), (1:7), and (1:9), using phosporic acid catalyst; 0,5%, 1,0%, and 1,5% (v/v).

The reaction is more pronounced at 50^0C to phosporic 0,5% (v/v) with the ratio of epoxide to ethylene glycol 1:1 (mol/mol), where the density of PU 1 is existed the highest among others, eventhough in the form of soy-polyol product the SP 3 has achieved high oxirane number compared to SP 1. But if the synthesis is decide at 70^0C, the best H_3PO_4 catalyst amount is 1,0% (v/v) for all ratio concentration of epoxide-glycol.

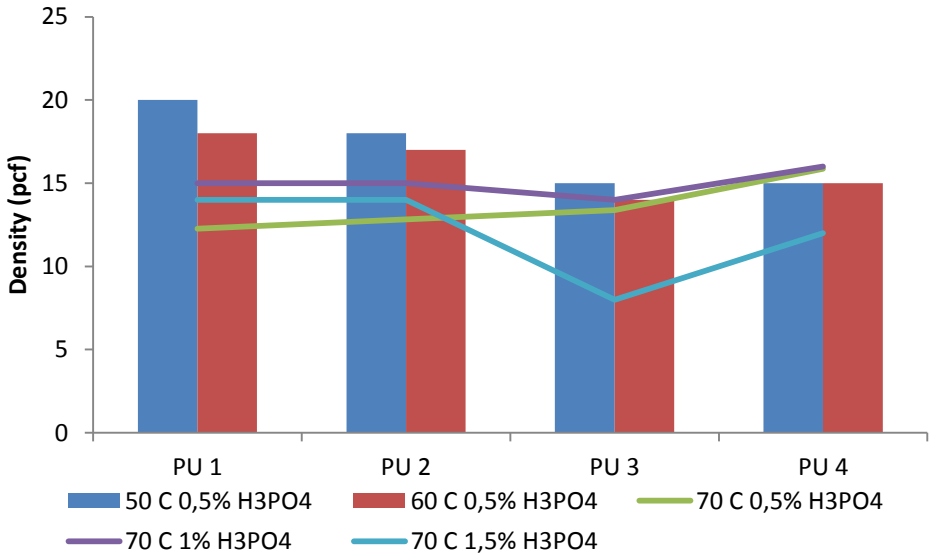

Figure 2. Catalyst concentration and temperature to density

4.2. Effect of Polyol and its blends on the properties of PU

Mechanical and physical properties PU foams are depends on functionality, equivalent weight, and hydroxyl value of polyols. The equivalent weight of polyol in common is 13,5 grek as compared to polyol 1 was 208 grek; polyol 2 was 181 grek, and polyol 3 was 122 grek. The equivalent weight is one among others parameter should be consider for the preparation of PU. The value are correlates to PU density which declined as value increased. The polyurethane from soy-polyol 4 labeled as PU 4 is found more rigid than PU 1, PU 2, and PU 3.

Aligned to the research founding, the foam manufacture using temperature 70^0C has the optimum density compared to 50^0C and 60^0C. Among temperature 70^0C, the optimized density has occured either using 0,5%, 1,0%, and 1,5% (v/v), they are categorized as flexible foam specially in the form of PU 2.

4.3.The Effect of Chain extender on PUF Impact Resilience

Water reacts with isocyanates forming carbon dioxide as chemical blowing agent, which in most cases chemical blowing agents such water are used in lower quantities compared to physical blowing agents. The ball used in this experiment was weighted 46 gr for ball 1 and 27 gram for ball 2. The addition of EG in PU's formula (4,26; 21,3; 29,8, and 38,34 mL) are respectively for PU 1, PU 2, PU 3, and PU 4 which made different responses to foam elasticity, as summarized in table 3. The resilience impact of PU are viewed as function of chain extender, phosporic concentration, and temperature of reaction to the bouncing responses. Phosporic react directly with oxirane group. Reduction oxirane content was observed with 1,5% (v/v) of phosporic. The result of PU 2 is classified as flexible while for PU 1 and PU 4 are less flexible. The fact for PU 1, the ethylene glycol addition of approximately 4,26 mL to either 1,0 % and 1,5% (v/v) of H_3PO_4 showed no bouncing effect during the test. But the compression set test using 1,5% (v/v) H_3PO_4 as for an advanced test has proven is the best amongst.

Table 3. The Impact Resilience of PU

Type of PU	Elasticity to Ball Bouncing (%)			
	H3PO4 1% (v/v)		H3PO4 1,5% (v/v)	
	Ball 1	Ball 2	Ball 1	Ball 2
PU 1	nb	nb	70,42	nb
PU 2	7,04	9,39	70,42	3,36
PU 3	3,36	nb	8,39	3,36
PU 4	1,3	3,91	70,42	3,91

[a]nb is indicated as the ball is not bouncing. Ball 1:46 gr, ball 2: 27 gr

[b]PU Elasticity (%) = $\{100 - \frac{(initial\ height - foam\ height\ of\ bouncing\ impact)}{initial\ hegiht}\}$x 100%

4.4.The Effect of Formula to Cellular Morphology

The mechanical property of a cellular material are mainly depend on density. The cellular images

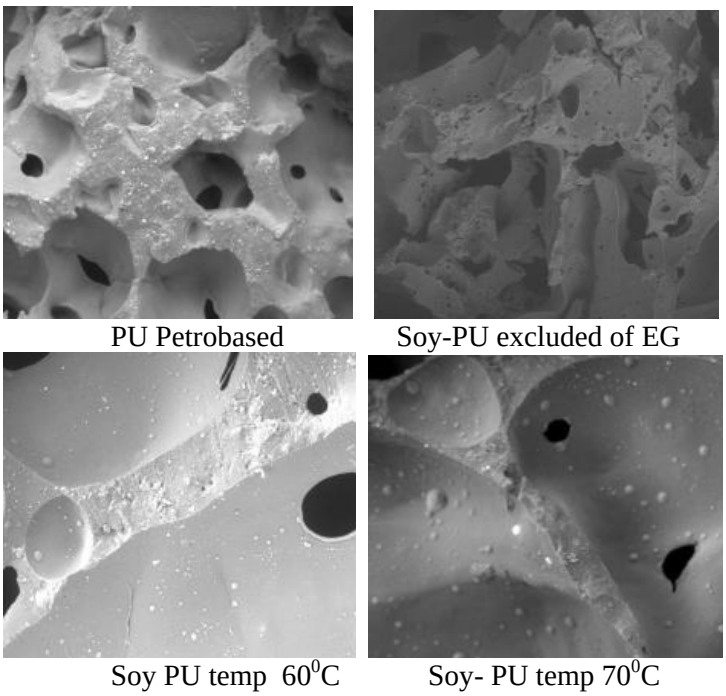

Figure 3. The SEM of PU

using SEM reveals the foam cells are slightly narrow with the enhancing temperatures. In this reaction, water molecules are as chemical blowing agent, it generates CO_2 on reaction with TDI isomers, resulted the evolution of heat. The temperature is effected to the volume of blowing gas in mixtures which exceeds its solubility limits and bubble nucleation. The SEM result has illustrated differentiate images of temperature effect during epoxide processing as previously designed was 50^{0}C, 60^{0}C and 70^{0}C. SEM images like those shown in figure 3 were examined and the average cell diameter are 0,1 mm for 50^{0}C; 0,5 mm for 60^{0}C; and 0,5 mm for 70^{0}C. The size difference between the foams is not statistically significant. The cellular images of PU without EG was vagued where at the initial state most of the cell faces are broken. Thus the SEM study and cell size, will only change

slightly suggested to the pathway from partial SP 1 to PU 1 with temperature 70^0C, and thus the observed foam mechanical properties are unlikely to be due to cell sizes.

Quite obvious and comparable using petroleum based polyol to soy based polyol with the same composition, it slightly higher mechanical strength is due to higher crosslink density,which results in a higher hard segment content. The cell size are reveals more larger this could be effected to the exceeds water content [15].

4.5.Polyurethane Foam Water absorption behavior

The water absorption behavior of PU prepared from soy-polyol using phosporic catalyst, and sulfuric acid was used as for comparative. The hydrophobic nature of long carbon chain as well as ester linkage of PU in the synthesis, are much comparable of using sulfuric acid to phosporic acid. The amount of H_3PO_4 1,5% (v/v) is more water absorbance than 1,0% (v/v). In general H_3PO_4 catalyst in PU synthesis resulted more water absorbance than sulfuric catalyst.

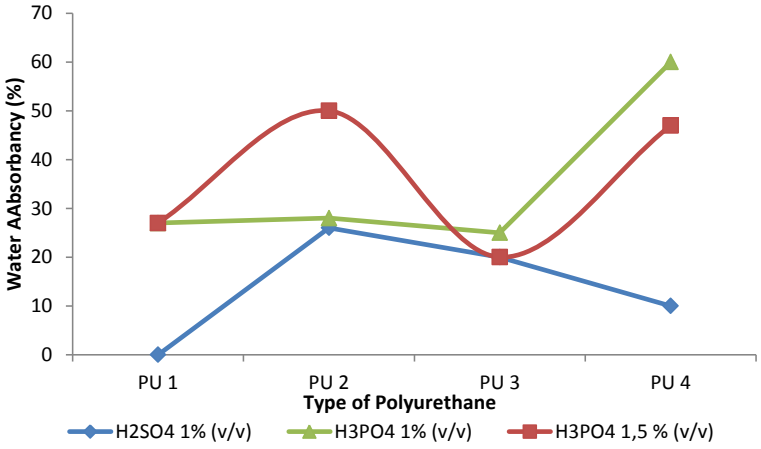

Figure 4. The polyurethane water absorbancy

[a] The foam is classified to the differentiation concentration of acid catalysis of H_3PO_4, H_2SO_4 was used as for the comparative

[b] H_2SO_4 1 % (v/v) is failed to react because of gel formation

4.6.Compression set of Polyurethane Foam

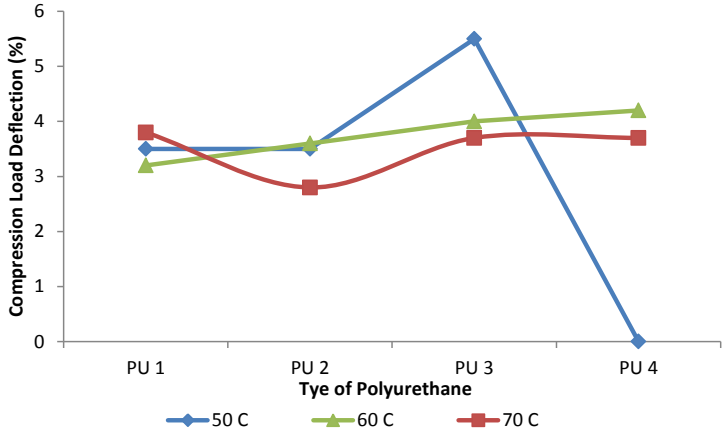

Figure 5. Polyurethane Foam Compression result

The foam characterization to percentage of compression set test has justified the foam flexibility value are recovered after compressed between two metal plates at controlled conditions: time and room temperature. The foam PU synthesized at 70^0C were discovered as the best among other foams because of its lowest value to recover to its original thickness.

Conclusion

On this study we found formula of making PU with optimum mechanical properties. Using phosporic acid catalyst, ratio of chain extender to epoxide, temperature in the PU synthesis has made responses to foams during characterization which then categorized into flexible and less flexible. The formula has much related to density, impact resilience, compression set, water absorbency behavior, and cellular morphology.

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