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Comparison Study on Morphology and Mechanical Properties of Starch, Lignin, Cellulose – Based Polyurethane Foam

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Abstract. In this research, new hybrid bio-polyurethane foam was fabricated by reacting with biomass such as lignin, starch and cellulose. Hybrid bio-polyurethane foam was prepared by reacting Toluene Diisocyanate 80 (TDI 80) and Polypropylene Glycol 2000 (PPG 2000), and then the variation of biomass was added to the reaction. Hybrid bio-polyurethane foam was characterized its tensile strength, indentation load deflection and airflow. Mechanical properties measurements indicated that the highest tensile strength was obtained polyurethane (PU)-lignin foam meanwhile the highest hardness was obtained PU-cellulose foam. Airflow test showed that PU-cellulose foam has more closed cell than PU-lignin and PU-starch. The morphology of foam was investigated using field emission scanning electron microscopy (FE-SEM). The SEM showed that starch has a smaller pore surface than other biomass. The cell morphology of hybrid biomass based polyurethane foam were closed with some particle stick onto the cell wall.

INTRODUCTION

Polymer material is widely applied in everyday human life, in the automotive sector to household appliances [1]. Every year, natural and synthesis polymer technology has rapid development in the industrial. Extensive application and relatively low manufacturing costs have triggered new research studies in polymer material, one of them is polyurethane (PU) which is generally produced into foam on various furniture [2]. PU is known as a multifunction material because it has unique characteristics, including lightness, high toughness, high elongation, high abrasion resistance, easy to processing, and relatively inexpensive. This makes PU not only applicable as foam, but also can be applied widely in the world of packaging, textiles, and biomedicine.

Advances research in the fields of chemistry and technology make the use of polyurethane increasingly commercial [3–5]. The industry's interest in PU foam continues to increase because it can be used as a varied product [6]. The basic ingredients for making polyurethane consist of diisocyanates and polyols. In general, there are two stages of making polyurethane, namely the reaction of diisocyanate with polyol then reacted with a chain extender [2]. An important characteristic possessed by PU foam is having a cavity with an open cell that is interconnected. This structure plays an important role in controlling mechanical and thermal properties [7]. Air-filled pores make PU foam elastic and isolator. The elastic properties can be modified to become rigid so that it can be applied as a car headliner. Headliners are mounted in the ceiling in a car with the aim of decoration, temperature insulation, soundproofing, and passenger protection.

Polyol commonly used in synthesizing PU foam is usually obtained from a polyether or polyester group which is a product of petroleum derivatives. Polyether polyols have a smaller hydroxyl number (OH number) so that it is used to synthesize polyurethane flexible foam, while polyester polyols have a larger OH number so that the foam

produced will be more rigid[7][8]. This hydroxyl group can also be obtained from natural hydroxyl groups or commonly called biomass. The level of sales of a car cannot be separated from the cost factor. To produce a car headliner, modification of the properties of PU foam from flexible to rigid can be done by adding biomass material to PU foam. This biomass can be easily obtained in nature with a relatively cheap price, in addition biomass is a material that is easily degraded into a material that is more environmentally friendly [9]

Starch, lignin, and cellulose are biomass with natural polymer chains that are rich in hydroxyl groups, which can be used as polyols in the reaction of polyurethane synthesis [10]. Although this research on biomass has been done by many people, research related to biomass to the mechanical properties still needs further research. In this study, the addition of biomass material to PU foam to improve its mechanical properties. This study focuses on the effect of biomass types on the increasing mechanical properties of polyurethane-foam and its compatibility review by studying morphology of PU foam. The best biomass type will increase the number of crosslinks formed so that it can increase mechanical properties.

EXPERIMENTAL

Materials

Polyol polypropylene glycol (PPG) 2000 Voranol 8010 from PT. DOW Indonesia, toluene diisocyanate (TDI) 80 Cosmonate T-80 from Mitsui Chemicals and SKC Polyurethane Inc., methylene chloride (MC) with 99.9% purity from Samsung Fine Chemical Co. Ltd., NIAx amine catalyst A-230 from Momentive Performance Materials, NIAx Silicone Surfactant from OSI Specialties Singapore PTE Ltd., tin catalyst KOSMOS 29 from Evonik Industries AG, and Kraft lignin, starch, and cellulose from Sigma Aldrich.

Preparation of PU foam

As much as 600 ml of PPG 2000, 354.80 ml of TDI 80, and 36 ml of MC as a solvent, then 25.68 ml of water and 0.6 of ml amine catalyst as blowing agent, 9.9 ml silicon surfactant, and 1.8 ml of tin catalyst as gelling agent are added. For independent variables used in this study are three types of biomass material in the form of lignin, starch, and cellulose powder as much as 21 grams.

Material Characterization

Tensile testing to study mechanical behavior of ultimate tensile strength (UTS) and elongation, then thermal testing to study changes in the temperature of degradation due to biomass addition. Morphological observations confirmed by chemical composition testing are useful to determine the compatibility between polyurethane foam and biomass. After that, density testing is done which has a relationship with stiffness.

Field Emission Scanning Electron Microscopy (FE-SEM) functions to analyze the size and shape of foam by looking at morphological observations. The tool used was the FEI Inspect F50 with a magnification of 100-5000x at the Center for Material Processing and Failure Analysis (CMPFA), Universitas Indonesia. Sample preparation is done by dipping the sample into liquid nitrogen so that it becomes brittle then the sample is broken. The sample is broken in brittle conditions so there is no mechanical influence during fracture.

Density and tensile testing were carried out by PT Inoac Polytechno Indonesia. Density testing is carried out based on the JIS K 6400 standard. Then, Universal testing machine (UTM) tool The Gotech AI-7000S draws dogbone samples to fractures with a constant speed (500 ± 50) mm per minute at room temperature. In addition, indentation load deflection (ILD) was also carried out. ILD is a hardness measurement of a foam that is measured by the amount of pressure needed to indentify a 4 inch thick foam up to 25% thick using a 50 square inch indenter. In addition to pressing up to 25% thick foam there is also ILD testing carried out up to 65% thick foam called ILD65. The thermal resistance testing used in this study is differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

RESULT AND DISCUSSION

Morphology hybrid bio-polyurethane foam

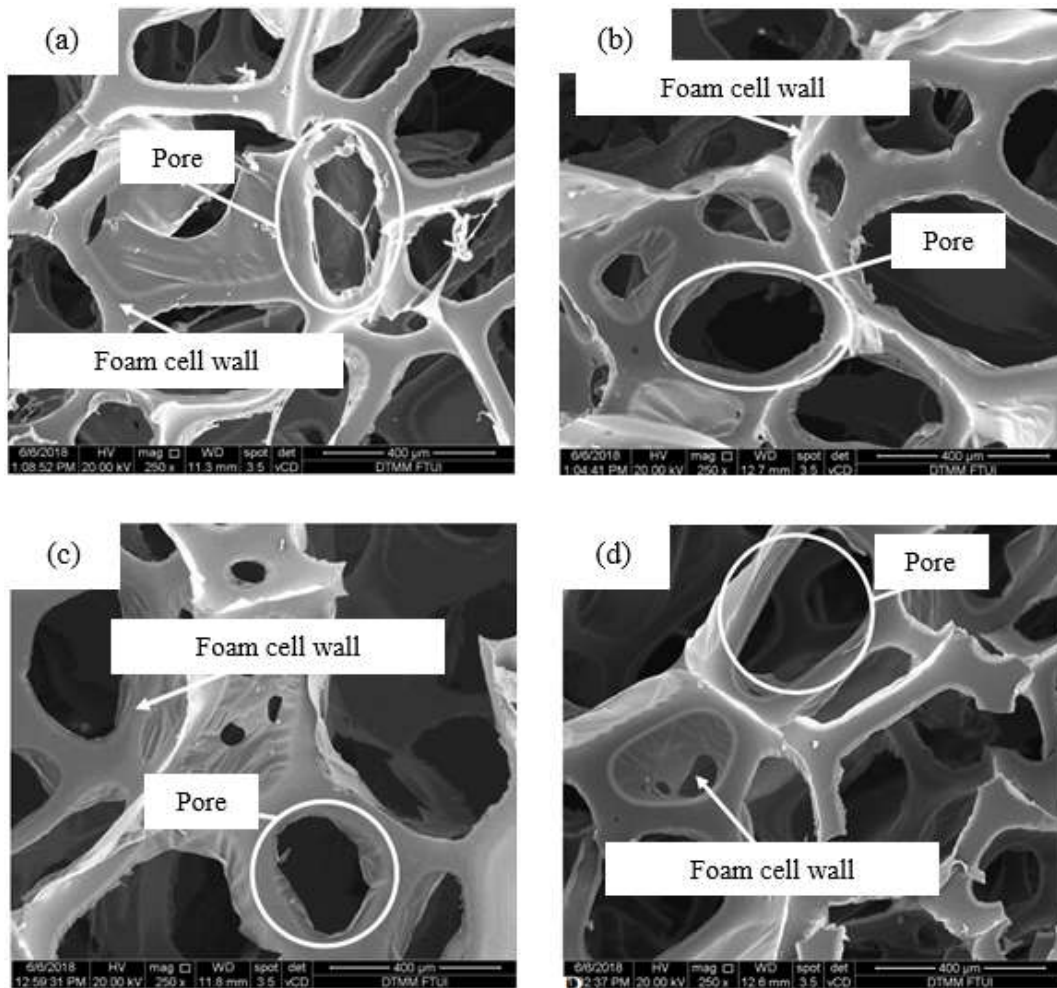


FIGURE 1. Morphology of samples (a) PU-virgin, (b) PU-lignin, (c) PU-starch, and (d) PU-cellulose with 250x magnification

The use of FE-SEM was carried out to facilitate observation and conclusions in terms of morphology. Morphological images can be seen in *Fig.1*. Observation of the morphological results found in *Fig.1* uses a 250x magnification, shows that the pores formed are in the form of open pore structures, this is in accordance with the results of research conducted by Alfani et al (1998) [11] which explains that by adding starch to polyurethane foam, the structure will result in an open cell. Henceforth, it is necessary to study the effect of starch concentration on the pore size of papa polyurethane foam. On the other hand research on the effect of cellulose concentration on pore size has been studied by Saz-orosco (2015) [12] that the increasing concentration of wood flour will decrease cell diameter and increase cell density.

Density and Mechanical Properties of hybrid bio-polyurethane foam

From the result of tensile strength as in *Fig.2a*, PU-virgin has 0.06 MPa. That is the standard number from the PU. After being given treatment in the form of lignin addition, got the number 0.08 MPa. There is an increase of 0.02 MPa which means it gives a positive result. While in PU with the addition of starch, the number 0.059 MPa is obtained, which means that there is a 0.01 reduction of PU-virgin. It can be concluded that additives in PU-virgin

can provide flexibility that is better than starch. Then in PU foam given cellulose gives the number 0.062 MPa. There is an increase of 0.02 MPa. Of the four samples, the best results were given to PU-Lignin with a number of 0.08 MPa. A significant increase indicates that lignin provides the best flexibility among the others.

Figure 2b shows a percentage diagram of elongation from each sample. PU-virgin has 196%. That is the standard number from the PU. PU-lignin produces elongation of 200%. There is a 4% increase in PU-virgin. In PU-starch, there was 202%, which showed a 6% increase in PU-virgin. And the last sample is PU-cellulose which only gives 106%. A pretty drastic reduction of 90%. From these results indicate that cellulose gives a good density value but not in terms of elasticity. In this test, the best results were obtained by PU-starch with 202% which was then followed by PU-lignin with a yield of 200%. These results provide a positive value that they have better elasticity than the additives found in PU-Virgin.

The results of the tear strength shown in Fig.2c provide an overview of the ductility of PU foam. PU-virgin produces a number of 0.048 MPa, which is a standard benchmark for other samples. In PU-lignin, the figure is 0.053 MPa. A positive result with an increase of 0.005 MPa. While in PU-starch, the number is 0.052 MPa. An increase of 0.004 MPa. It also gives us positive results. Then the PU-cellulose results obtained at 0.037 MPa. A decrease of 0,011 MPa from PU-virgin which also gives the conclusion that cellulose provides an increase in terms of rigidity, not ductility. The best result of this tear strength is PU-lignin with a number of 0.053 MPa which indicates that lignin can provide good ductility and flexibility than additives in PU-virgin.

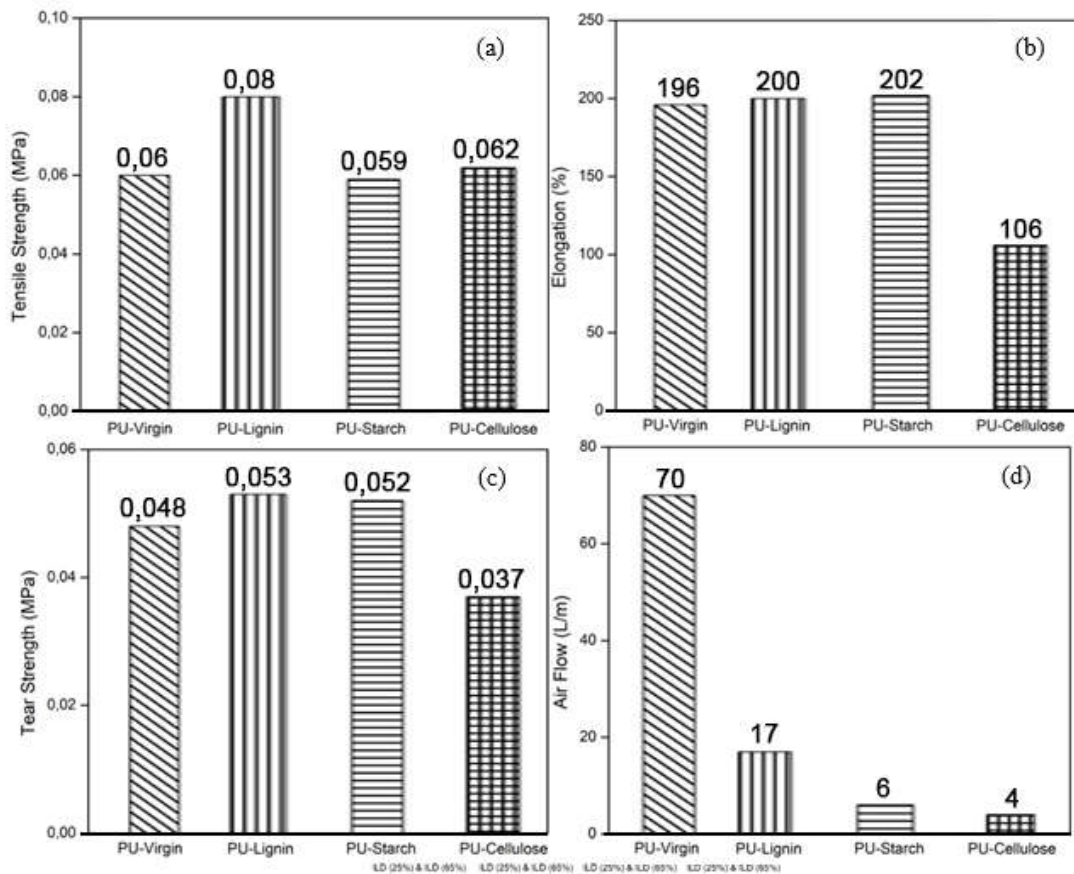


FIGURE 2. Comparison Chart of (a) Tensile Strength, (b) Elongation, (c) Tear Strength, (d) Air Flow

The air flow test is carried out by placing polyurethane foam that has been cut with certain dimensions of the air blower and then exhaled air into polyurethane foam. Barometer showed how much air you can get flowing through foam. This value generally correlates with the level of cell openness in foam. The higher air flow means the foam has more open cell. The results of measuring water flow are as shown in Fig.2d. The highest air flow is owned by

PU virgin which is not added by biomass is equal to 70 L/min, then for the lowest air flow is owned by PU-cellulose which is 4 L/min. This result indicates that PU-cellulose has most number of closed cells compared to other samples, then this is probably related to its tear strength and elongation.

CONCLUSION

In the results of morphological tests, it is seen that there are small particles which may be particles of biomass that are attached to the foam cell wall but look not tightly attached. This is indicated by the gap between these particles and the cell wall of polyurethane foam. Biomass based polyurethane foam has better mechanical properties compared to polyurethane virgin foam. Curing process can increase the amount of physical crosslink in the form of hydrogen bonds and chemical crosslink in the form of covalent bonds. PU-lignin has the highest UTS of 0.06 MPa and the highest tear strength of 0.053 MPa. PU-cellulose has a highest stiffness by elongation of 106%, also the highest hardness by ILD25 of 0.0039 MPa and ILD65 of 0.0084 MPa.

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