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Manufacturing Polyurethane Slag from Composite using Polyol from Polyethylene Plastic Waste

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This research aims to develop polyurethane foam using recycled PET (Polyethylene Terephthalate) and HDPE (High-Density Polyethylene) plastic bottles as substitutes for polyol. Waste PET bottles were recycled through a glycolysis process to produce BHET (bis(hydroxyethyl) terephthalate), utilized as a polyol substitute in polyurethane foam production. The foam was synthesized by reacting polyol with Methylene Diphenyl Diisocyanate (MDI), with variations in the composition of distilled water as a blowing agent, silicone as a surfactant, and steel slag (10%, 10%, 10%, and 60%) to enhance mechanical properties. Four polyurethane foam samples were tested, resulting in rigid, flexible, and semi-rigid foams, depending on the formulation. Sample 1 demonstrated a compressive strength of 0.225 MPa, Young's modulus of 0.0139 MPa, yield strength of 0.174 MPa, and density of 0.11 g/cm³. Sample 2 exhibited a compressive strength of 0.18 MPa, Young's modulus of 0.0109 MPa, yield strength of 0.117 MPa, and density of 0.06 g/cm³. Sample 3 had the lowest compressive strength (0.02 MPa), Young's modulus (0.00079 MPa), yield strength (0.0092 MPa), and density (0.09 g/cm³). Sample 4 recorded a compressive strength of 0.12 MPa, Young's modulus of 0.0116 MPa, yield strength of 0.0901 MPa, and density of 0.04 g/cm³. Sample 1 exhibited the highest mechanical performance, while Sample 3 showed the lowest. These results indicate that polyurethane foam with optimal compressive strength, Young's modulus, yield strength, density, and flexibility can be produced, meeting the requirements of SNI (*Standar Nasional Indonesia*) Standard 0111-2009.

Keywords: Composite; polyol; polyurethane foam; recycle; steel slag.

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Introduction

Throughout 2022 Indonesia will produce 19.45 million tons of landfill waste. Waste is one of the biggest problems for all of us, and waste is leftover material from our activities that have no use. Therefore, it must be managed [1], becoming a serious challenge with the ever-increasing population growth in Indonesia. In 2022, Indonesia's population will reach 270.2 million people [2], which means the potential for waste produced will reach 33 million tons. Based on these figures, waste contribution is dominated by households, namely 39.63%, followed by

waste originating from commerce at 21.07% and the market at 16.08%. Based on type, most of the national waste generation is food waste with a proportion of 41.55%, plastic waste with a proportion of 18.55%, waste in the form of wood/twigs 13.27%, paper/cardboard 11.04%, metal 2.86%, cloth 2.54%, glass 1.96%, rubber/leather 1.68%, and other types of waste 6.55% [3].

Steel slag (SS) is a by-product of the steelmaking industry and is considered industrial waste often sent to landfill sites. Efforts have been made to find new potential applications for SS to reduce its disposal. Rigid polyurethane foam (RPUF) is a polymer material with

advantages such as low density and high specific strength but a high fire risk. Various strategies have been explored to develop flame-retardant RPUFs, including reactive-type and additive-type methods. However, the high loading of additive-type flame retardants often leads to a decrease in physical-mechanical behavior. This research aims to expand the application of steel slag waste by modifying it with silane containing phosphorus and incorporating it into RPUF to increase fire resistance. The effect of modified steel slag (MS) on the compression strength, thermal stability and fire resistance of RPUF was investigated [4].

Plastic is an important material in everyday life because it is light, waterproof, anti-rust, heat and electrical insulator, and affordable. This causes global plastic production to reach 130 million tons per year [5], with 40% of consumption focused on plastic packaging. The main challenge for plastics is sustainable management and disposal to reduce environmental impact [6]. PET (Polyethylene Terephthalate) and HDPE (High-Density Polyethylene) plastics are widely used in industry. PET is used for bottles of water, cooking oil, juice, chili sauce, medicine and cosmetics, while HDPE is used for packaging liquid products such as detergent and oil [7]. Plastic waste management must involve the 3R principles: reuse, reduce and recycle [8]. Recycling, especially for PET and HDPE, can be carried out through various methods such as glycolysis, *methanolysis*, hydrolysis, ammonolysis, and *aminolysis* [9].

The PET and HDPE glycolysis process is a commonly used depolymerization method due to its simplicity, although it requires a catalyst. Research has increased the efficiency of glycolysis with various catalysts such as metal acetate, titanium phosphate, superacid, metal oxide, and sulfate [10]. Based on previous research, the glycolysis process in HDPE (High-Density Polyethylene) waste is carried out using a catalyst in the form of metal acetate, including zinc, tin, cobalt and manganese acetate. The research results show that zinc acetate, with a composition of 0.5% of the weight of the plastic used, is a catalyst capable of producing BHET (bis(hydroxyethyl)terephthalate) with the best performance [11]. This research shows that the zinc acetate catalyst is most effective in producing BHET (bis(hydroxyethyl)terephthalate) from HDPE, with varying ratios, such as 1:2, 1:4, 1:5, and 1:6, the optimal mole ratio of plastic and solution glycol 1:4. [12].

Polyurethane, discovered by Otto Bayer, can be converted into polyurethane foam (PUF), which is widely used as an insulation material and in the automotive industry [13]. PUF production usually uses polyols from petroleum, which causes environmental problems due to the use of non-renewable energy resources [13]. To overcome this and reduce plastic waste, recycled polyols from PET and HDPE were developed through depolymerization with glycolysis. This polyol can be used as a polymer component in the manufacture of PUF, integrating plastic recycling to reduce environmental impact and provide added value to plastic waste management.

Making polyurethane foam involves isocyanate (MDI), blowing agent, and surfactant as the main raw materials. As an alternative to dangerous blowing agents

such as HCFCs and CFCs, Kim Roland developed a water-based blowing agent in 2002 [14]. This blowing agent plays an important role in forming the foam structure by producing CO₂ gas when it reacts with MDI, forming air bubbles which provide porosity to the foam. The use of Methylene Diphenyl Diisocyanate (MDI) can produce various types of polyurethane foam, including rigid, semirigid and flexible, with the right blowing agent formulation influencing the mechanical properties of the polyurethane foam. Each type of foam has different applications according to its characteristics. The differences in the physical properties of these three types of foam depend on variations in molecular weight, polyol functionality, and isocyanate functionality used.

Polyurethane foam has a cell structure which can be closed cell or open cell, which affects its properties. Rigid foam uses MDI to form a rigid structure, suitable for thermal insulation, wall panels and construction. Semirigid foam uses specially formulated MDI to produce medium hardness foam, used in cushioning and structural components. Flexible foam uses MDI for soft and elastic foam, often used in mattresses, soft furniture, and other comfort products. For applications as fire-resistant insulation, polyurethane foam can be modified with halogen compounds [15]. This combination of properties makes polyurethane foam very versatile in industry and construction.

Polyurethane foam is very versatile and is widely used in industry and construction. Its uses include thermal insulation, electrical insulation, sealants, automotive foams, and furniture materials [13]. Polyurethane from PET and HDPE waste is the raw material for making PUF as an effort to manage plastic waste in a more environmentally friendly way, with innovation in the use of water-based blowing agents [16].

The use of steel slag as an additive in the production of polyurethane foam significantly improves the mechanical properties of the material. Steel slag, a byproduct of steel production, contains minerals that increase the strength and hardness of polyurethane foam. Its addition can increase the density and modulus of elasticity, making the foam stiffer and more pressure resistant. Steel slag also improves the cellular structure of the foam, distributes cells more evenly, and reduces pore size, which increases mechanical strength and thermal insulation. However, the amount of steel slag must be optimized, because excess can reduce elasticity and make the foam brittle. Further research is needed to determine the optimal composition that maximizes benefits without compromising foam flexibility and durability.

Thus, these findings are a basis for consideration for conducting further research regarding the Effect of Adding Slag Composition on the Structure, Morphology, Density and Compressive Strength of Polyurethane Foam Using Polyethylene Terephthalate (PET) and High-Density Polyethylene (HDPE) Plastic Waste. This research aims to explore the impact of the use of MDI and Slag on the mechanical properties of polyurethane foam, with an emphasis on the use of plastic waste as raw materials for shoe industry applications. With this research, it is hoped that it can provide additional insight into the efficiency and sustainability of polyurethane foam production by considering environmental aspects.

I. Material and methods

The first process is the process of making polyol from PET plastic waste and other types of plastic waste such as HDPE using the glycolysis method.

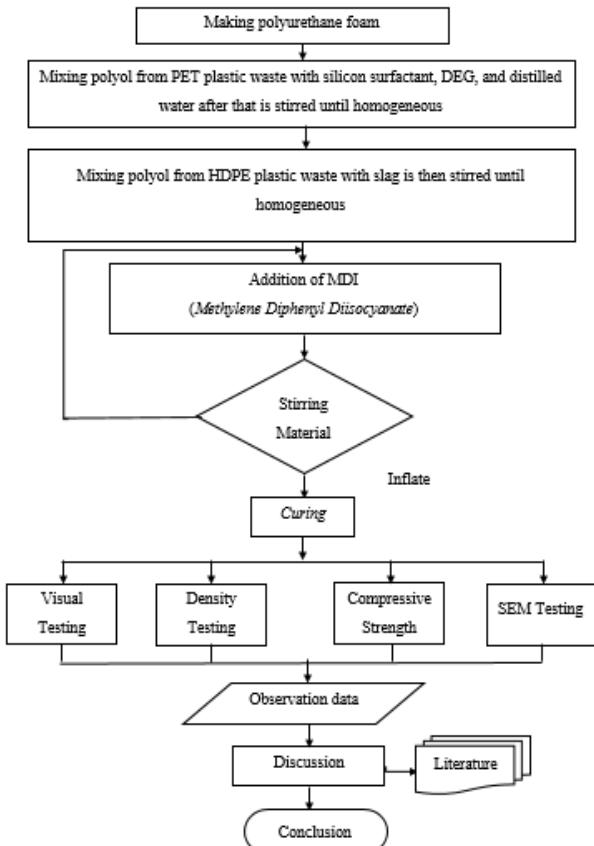
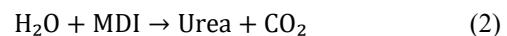
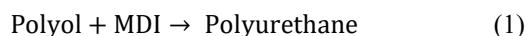


Fig. 1. Flow diagram for making polyurethane foam.

Plastic waste plastic bags and straws are cut into small sizes, then used to make polyol material. The process of making polyol from PET plastic waste begins by mixing plastic bottle pieces with zinc acetate in a proportion of 0.5% of the amount of PET plastic waste used. Next, diethylene glycol was added to 1% of the weight of 100% PET polyol. The mixture of ingredients was heated to a temperature of 275°C in a muffle furnace for 1 hour 30 minutes, then left to stand at room temperature. Plastic waste plastic bags and straws are cut into small sizes, then used to make polyol material. The process of making polyol from PET plastic waste begins by mixing plastic bottle pieces with zinc acetate in a proportion of 0.5% of the amount of PET plastic waste used. Next, diethylene glycol was added to 1% of the weight of 100% PET polyol. The mixture of ingredients was heated to a temperature of 275°C in a muffle furnace for 1 hour 30 minutes, then left to stand at room temperature. The next step involves making rigid Polyurethane Foam (PUF) by mixing 100% of the weight of PET polyol, for making semirigid Polyurethane Foam (PUF) by mixing polyol from another type of plastic HDPE with distilled water 1.32% of the weight of polyol, then adding silicon surfactant 4 % of polyol weight. This mixture is stirred until homogeneous and poured into molds. The next process involves adding Methylene Diphenyl

Diisocyanate (MDI) with 1.7% of the weight of the pre-PU that has been produced. All ingredients are then stirred using a spatula until the curing process occurs, and the results are left to harden. The characterization using visual test, density test, SEM and compressive strength test.

The process of making polyurethane foam using PET as polyol is carried out using the glycolysis method. In the glycolysis method, PET that has been cut into pieces up to 4 mm in size is dissolved with diethylene glycol (C₄H₁₀O₃) and zinc acetate (Zn(CH₃CO₂)₂) by heating 275°C in a muffle furnace for 1.5 hours. C₄H₁₀O₃ acts as a glycol solvent, while Zn(CH₃CO₂)₂ acts as a catalyst which functions to accelerate the rate of the glycolysis reaction. Apart from that, zinc acetate can produce depolymerization products with a yield of 78% [41]. From the 2 samples that were made, the volume of polyol produced was all constant with the same weight of 63.7 grams for PET, while for HDPE it weighed 97.62 grams. In the process of making polyurethane foam composites, polyol compounds react with several additional ingredients. Aquadest, as a blowing agent, is responsible for developing foam [21]. Diethylene glycol is present as an anti-foaming agent, while silicon surfactant acts as a surfactant which reduces surface tension and helps the dissolution of distilled water with MDI [22]. MDI (Methylene Diphenyl Diisocyanate) acts as an isocyanate, functioning as a bridging agent that binds polyols and forms a polymer network structure [21]. Steel Slag meanwhile, is used as reinforcement against high temperatures [23]. In the process of making polyurethane foam composites, several phenomena occur. For example, the formation of smoke and heat during the expansion process. This is caused by an exothermic reaction between distilled water and MDI which produces carbon dioxide gas (CO₂) and amide as by-products. This reaction releases stored energy, which is then released in the form of heat and CO₂ gas. This reaction is the basis of the formation of polyurethane foam:



The composition used in making polyurethane foam includes several main and additional ingredients, each of which has an important role in determining the final characteristics of the foam. Based on Table 1, the basic plastics used include PET (Polyethylene Terephthalate) and HDPE (High-Density Polyethylene).

Table 1 shows the composition of polyurethane foam polyols that use PET and HDPE as plastic base materials. In this table, we see that although PET starts with 40 grams, the weight of the resulting polyol is 43.63 grams, while for HDPE, an initial weight of 16 grams results in a polyol weight of 102.73 grams, and there is a residual plastic weight of 18.21 grams. Some of the reasons why PET with an initial weight of 40 grams produces a relatively small polyol weight compared to HDPE can be caused by several chemical reaction factors. PET reacts with diethylene glycol (DEG), zinc acetate (Zn(A)) to produce PET polyol. HDPE reacts with DEG, zinc acetate (Zn(A)), and SnO₂ but produces larger plastic residues due to degradation reactions that

may not be completely efficient. Thus, the differences in reaction yield and plastic residue between PET and HDPE are likely due to differences in their chemical structures, polymerization reaction efficiencies, and different reaction conditions.

Once the polyol is obtained, stir it so that all the compounds are homogeneous, then wait for it to expand for about 30 seconds and cover the Mold with another Mold as a lid so that the foam does not overflow when it expands. In making foam, it produces polyurethane foam which produces rigid, semi-rigid and flexible foam according to its texture.

II. Results and Discussion

In making polyurethane foam composites, the addition of steel slag aims to increase strength at high temperatures. Steel Slag is integrated in the process to produce a polyurethane foam composite that has resistance to high temperatures, as well as to achieve the desired mechanical properties and morphological structure. The level of steel slag composition introduced affects the strength and density of the foam slag composite. The greater the proportion of steel slag used, it will affect the compressive strength and density of the resulting slag foam composite [25]. However, it is important to remember that these properties are also influenced by the composition of the surfactants involved in the process. It is important to note that the results of variations in steel slag composition can be found in table 3, which gives an idea of how the composition affects the characteristics of the resulting polyurethane foam

composite.

In this strength test, the main aim is to analyse the strength of the polyurethane foam that has been made. Based on Table 3, there is a Young's modulus value for each composition, where the Young's modulus value can be obtained by looking for the slope of the resulting stress-strain graph. This test follows the ASTM D1621 standard, which states that the yield strength value can be obtained from the stress-strain graph by determining the peak point of bending. However, if the peak point of the curve is less visible, the offset method can be used. The offset method involves determining a point on the stress-strain graph that is a certain offset (for example 10%) from the stress axis, making it easier to determine an accurate yield point. In this study, the stress-strain graphs showed consistent results for the various samples tested. For the steel slag composition in Sample 1 of experiment 1 and Sample 2 of experiment 2, the use of the 10% offset method gave clear and accurate results regarding the yield strength point. The same applies to Sample 3 of experiment 1 and Sample 3 of experiment 2, which also used a steel slag composition of 10%. This stress-strain graph can be seen in Figure 2, which provides a visual illustration of how stress and strain interact at various points during testing.

In Table 3, that Sample 1 and Sample 3 with variations in steel slag composition of 10% and 10%, have different compressive strength, Young's modulus and yield strength values. Based on the literature, the greater the Young's modulus value of an object, the stiffer the object will be, so the compressive strength value of an object will be greater. Based on the literature, the size of the steel slag composition used will affect the compressive strength and density of the resulting slag foam composite. Figure 2

Table 1.

Polyol Composition of Polyurethane Foam

Plastic Type	Plastic Weight	DEG	Zinc Acetate (A)	SnO ₂ (B)	Polyol Weight	Residual weight of plastic
PET	40 gram	44.20 gram	0.5 %	-	43.63 gram	-
HDPE	16 gram	105.34 gram	0.5 %	1 %	102.73 gram	18.21 gram

Table 2.

Polyurethane Foam Composition

Sample	PET (A)	Other Polyols (B)	H ₂ O (pphp) (C)	SURF (pphp) (D)	DEG (pphp) (E)	MDI (pphp) (F)	Slag (pphp) (G)
Sample 1 (10.09 gr)	97.1 %	2% (HDPE)	4%	1.32%	1%	1.4	10%
Sample 2 (10.38 gr)	96.55 %	3% (HDPE)	4%	1.32%	1%	1.4	10%
Sample 3 (10.25 gr)	96.6 %	3% (HDPE)	10%	4%	3%	1.4	10%
Sample 4 (15.00 gr)	96.5 %	3% (HDPE)	1.7%	4%	3%	1.4	60%

Table 3.

Polyurethane Foam Results with Steel Slag Variations

Sample	Slag (pphp)	Compressive Strength (MPa)	Young's Modulus (MPa)	Yield Strength (MPa)	Density (gram/cm ³)	Density (% Error)	Foam Type
Sample 1	10%	0.225	0.0139	0.174	0.11	1%	<i>Rigid</i>
Sample 2	10%	0.18	0.0109	0.1170	0.06	5%	<i>Rigid</i>
Sample 3	10%	0.02	0.00079	0.0092	0.09	2%	<i>Flexible</i>
Sample 4	60%	0.12	0.0116	0.0901	0.04	7%	<i>Semirigid</i>

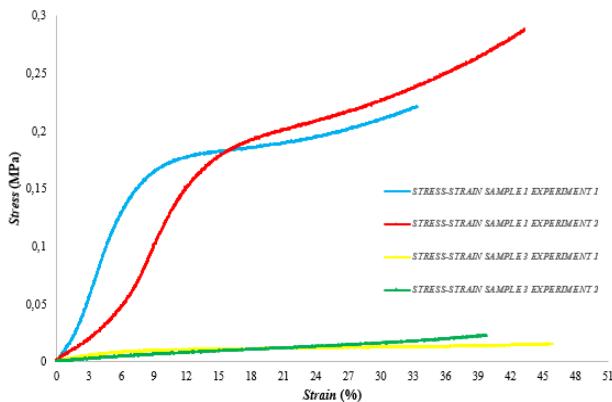


Fig. 2. Stress-Strain Graph of Steel Slag Composition Variations (a) 10% Sample 1 Experiment 1 and Experiment 2 and (b) 10% Sample 3 Experiment 1 and Experiment 2.

shows the stress strain graph and Young's modulus value for each sample. The Young's modulus value is obtained from the slope of the linear regression line (slope). The compressive strength value for Sample 1 in experiment 1 and Sample 1 in experiment 2 was 0.22 MPa and 0.29 MPa, the average obtained was 0.225 MPa. The compressive strength value for Sample 3 in experiment 1 and Sample 3 in experiment 2 was 0.02 MPa and the average obtained was 0.02 MPa. The Young's modulus value in Sample 1 of experiment 1 and Sample 1 of experiment 2 was 0.0158 MPa and 0.0120 MPa, the average obtained was 0.0139 MPa. The Young's modulus value in Sample 3 of experiment 1 and Sample 3 of experiment 2 was 0.00098 MPa and 0.0006 MPa, the average obtained was 0.00079 MPa. The yield strength values in Sample 1 of experiment 1 and Sample 1 of experiment 2 were 0.1741 MPa and 0.1739 MPa, with an average of 0.174 MPa. The yield strength values in Sample 3 of experiment 1 and Sample 3 of experiment 2 were 0.0097 MPa and 0.0088 MPa, with an average of 0.0092 MPa. If the Young's modulus data and compressive strength values are compared, the values are in accordance with the literature mentioned because the greater the Young's modulus of the foam, the greater the compressive strength value of the foam [24]. The composition of the steel slag used influences the increase in compressive strength and density of the resulting slag foam composite [25].

In Figure 3, it can be seen that in Sample 1, Experiment 1 and Sample 3, Experiment 1, with variations in the steel slag composition of 10% and 10%, had different compressive strength, Young's modulus and yield strength values. Sample 1 of experiment 1 and Sample 3 of experiment 1 with variations in steel slag composition of 10% and 10% were the best results because they had more significant compressive strength, Young's modulus, yield strength and graph shapes. The compressive strength value in Sample 1 in experiment 1 was 0.22 MPa. The compressive strength value in Sample 3 in experiment 1 was 0.02 MPa. The Young's modulus value in Sample 1 of experiment 1 was 0.0158 MPa. The Young's modulus value in Sample 3 of experiment 1 was 0.00098 MPa. The yield strength value in Sample 1 of experiment 1 was 0.1741 MPa. The yield strength value in

Sample 3 of experiment 1 was 0.0097 MPa. If Young's modulus data and compressive strength values are compared, it is in accordance with the literature that has been mentioned because there is a clear correlation between the two. Research by Lim in 2008 [24] showed that the greater the Young's modulus of polyurethane foam, the greater the compressive strength value. This shows that Young's modulus, as a measure of material stiffness, has a direct impact on the mechanical properties of polyurethane foam, including compressive strength. On the other hand, research by Tang in 2020 [25] found that the composition of the steel slag used in making polyurethane foam influenced the increase in compressive strength and density of the resulting slag foam composite. This shows that the addition of steel slag as an additional material can influence the mechanical and physical properties of polyurethane foam, including its compressive strength and density. Thus, integration of data from these studies provides a more complete understanding of the factors influencing the mechanical properties of polyurethane foam. This knowledge can be used as a basis for designing more optimal polyurethane foam formulations.

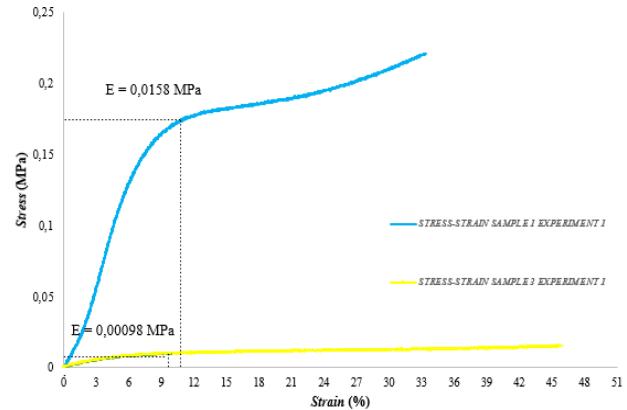


Fig. 3. Stress-Strain Graph of Steel Slag Composition Variations (a) 10% Sample 1 Experiment 1 and (b) 10% Sample 3 Experiment 1.

The stress strain graph with the steel slag composition for Sample 2 in experiment 1 and Sample 2 in experiment 2 is 10%. Meanwhile, the composition of steel slag in Sample 4 of experiment 1 and Sample 4 of experiment 2 is 60% which can be seen in Figure 4. In Table 3, it can be seen that in Sample 2 and Sample 4 with variations in steel slag composition of 10% and 60%, the values different compressive strength, Young's modulus, yield strength. Based on the literature, the greater the Young's modulus value of an object, the stiffer the object will be, so that the compressive strength value of an object will be greater. Figure 4 shows the stress strain graph and Young's modulus value for each sample. The Young's modulus value is obtained from the slope of the linear regression line (slope) on the stress-strain graph, which describes the relationship between stress and strain in an elastic material. Based on the ASTM D1621 standard, the yield strength value can be obtained from the stress-strain graph by determining the peak point of bending, where the material begins to experience plastic deformation. However, if the peak point of this curve is less clear or

difficult to identify, the offset method can be used as an alternative. The offset method involves determining a point on the stress-strain graph that is a certain distance (for example, 10%) from the stress axis, to more accurately determine the yield point of the material.

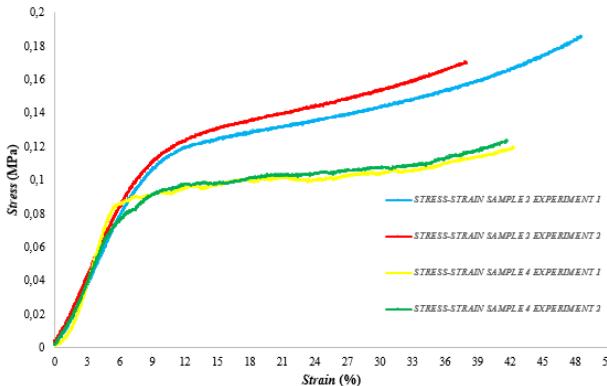


Fig. 4. Stress-Strain Graph of Steel Slag Composition Variations (a) 10% Sample 2 Experiment 1 and Experiment 2 and (b) 60% Sample 4 Experiment 1 and Experiment 2.

The compressive strength value in Sample 2 of experiment 1 and Sample 2 of experiment 2 was 0.19 MPa and 0.17 MPa, the average obtained was 0.18 MPa. The compressive strength value in Sample 4 in experiment 1 and Sample 4 in experiment 2 was 0.12 MPa and 0.12 MPa, the average obtained was 0.12 MPa. The Young's modulus value in Sample 2 of experiment 1 and Sample 2 of experiment 2 was 0.0107 MPa and 0.0111 MPa, the average obtained was 0.0109 MPa. The Young's modulus value in Sample 4 of experiment 1 and Sample 4 of experiment 2 was 0.0139 MPa and 0.0094 MPa, the average obtained was 0.0116 MPa. The yield strength values in Sample 2 of experiment 1 and Sample 2 of experiment 2 were 0.1149 MPa and 0.1192 MPa, with an average of 0.1170 MPa. The yield strength values for Sample 4 in experiment 1 and Sample 4 in experiment 2 were 0.0865 MPa and 0.0937 MPa, with an average of 0.0901 MPa. If the Young's modulus data and compressive strength values are compared, the values are in accordance with the literature mentioned because the greater the Young's modulus of the foam, the greater the compressive strength value of the foam [24]. The composition of the steel slag used influences the increase in compressive strength and density of the resulting slag foam composite [25].

In Figure 5, it can be seen that in Sample 2, experiment 2 and Sample 4, experiment 2, with variations in steel slag composition of 10% and 60%, had different compressive strength, Young's modulus, and yield strength values. Sample 2 of experiment 2 and Sample 4 of experiment 2 with variations in steel slag composition of 10% and 60% were the best results because they had more significant compressive strength, Young's modulus, yield strength and graph shapes.

The compressive strength value in Sample 2 in experiment 2 is 0.17 MPa. The compressive strength value in Sample 4 in experiment 2 is 0.12 MPa. The Young's modulus value in Sample 2 of experiment 2 was 0.0111 MPa. The Young's modulus value in Sample 4 of

experiment 2 was 0.0094 MPa. The yield strength value in Sample 2 of experiment 2 was 0.1192 MPa. The yield strength value in Sample 4 in experiment 2 was 0.0937 MPa. If the Young's modulus data and compressive strength values are compared, it is in accordance with the literature that has been mentioned because there is a clear correlation between the two. Research by Lim in 2008 [24] showed that the greater the Young's modulus of polyurethane foam, the greater the compressive strength value. This shows that Young's modulus, as a measure of material stiffness, has a direct impact on the mechanical properties of polyurethane foam, including compressive strength. On the other hand, research by Tang in 2020 [25] found that the composition of the steel slag used in making polyurethane foam influenced the increase in compressive strength and density of the resulting slag foam composite. This shows that the addition of steel slag as an additional material can influence the mechanical and physical properties of polyurethane foam, including its compressive strength and density. Thus, integration of data from these studies provides a more complete understanding of the factors influencing the mechanical properties of polyurethane foam. This knowledge can be used as a basis for designing more optimal polyurethane foam formulations.

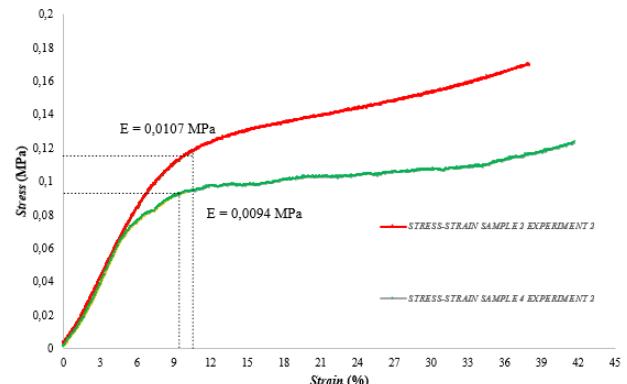


Fig. 5. Stress-Strain Graph of Steel Slag Composition Variations (a) 10% Sample 2 Experiment 2 and (b) 60% Sample 4 Experiment 2.

Based on the research results which state that the compressive strength of polyurethane foam from Sample 1, Sample 2, Sample 3, and Sample 4 does not meet the requirements of the SNI 0111-2009 standard for shoe applications, there is a discrepancy in the strength measurements required for shoe soles. The SNI 0111-2009 standard requires that polyurethane foam used for single-layer soles must have a minimum compressive strength of 5 N/mm², while for two-layer soles it must be at least 8 N/mm². From the reported research results, the compressive strength value of each polyurethane foam sample was below the specified standard value. This shows that the polyurethane foam tested cannot be considered to meet the compressive strength requirements required for shoe soles according to the SNI 0111-2009 standard. Further evaluation or modification to the polyurethane foam formulation may be necessary to increase the compressive strength to comply with the requirements required for shoe sole applications in applicable standards.

Density testing is a method commonly used to determine the specific gravity or density of a material. In the context of polyurethane foam manufacturing, density testing is important to understand how dense or light the resulting foam is. The density testing method is carried out by calculating the mass and volume of each specimen using Archimedes' principle. Archimedes' principle states that the buoyant force experienced by an object completely submerged in a fluid is equal to the weight of the fluid displaced by the object. In other words, when an object is immersed in a fluid (in this case, polyurethane foam), it pushes against a volume of fluid equal to the volume of the object. By measuring the volume of fluid displaced when an object sinks and knowing the mass of the object, the density of the object can be calculated. In density testing of polyurethane foam, the ASTM D1622 standard is used as a guideline for appropriate test procedures. The tool used is usually an analytical balance, such as AS 220.R2, which allows mass measurements with high precision. The composition of steel slag can affect the density value of polyurethane foam due to interactions between steel slag and other ingredients in the foam formulation. As previously explained, the addition of steel slag can reduce the foam density because the reaction between steel slag and MDI produces CO_2 gas which forms pores in the foam. When steel slag is used in larger quantities, the chemical reactions that occur in the metal foam manufacturing process can produce more gas. These gases are then trapped in the metal foam structure, increasing its porosity, which is the amount of empty space within the structure. Due to higher porosity, the density of the foam will decrease because the actual volume of the foam remains the same but contains more air or gas. Therefore, the more steel slag used in the metal foam manufacturing process, the lower the resulting foam density. Thus, density testing is an important step in the characterization of polyurethane foam, while the steel slag composition plays a role in determining the final density of the foam by influencing the formation of pores and its physical structure. In Figure 6, it can be seen that the greater the steel slag composition used can affect the foam density value. Foam with a steel slag composition of 10% in Sample 1 had an average density value of 0.11 gram/cm³ and the density error percentage obtained was 0.47%. The steel slag composition of 10% in Sample 3 has an average density value of 1.13 grams/cm³ and the density error percentage obtained is 2%. The data obtained is in accordance with existing literature, where the greater

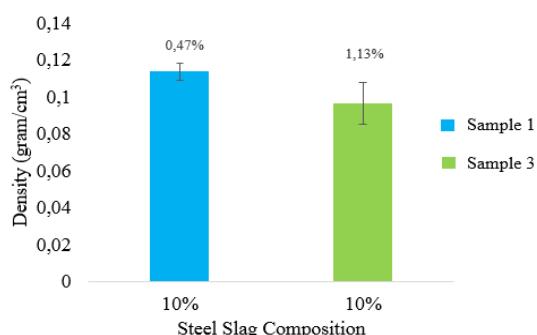


Fig. 6. Graph of %Error Density Values with Steel Slag Composition (a) 10% Sample 1 and (b) 10% Sample 3.

the steel slag composition used, the density value decreases [25]. In Figure 7, it can be seen that the greater the steel slag composition used can affect the foam density value. Foam with a steel slag composition of 10% in Sample 2 had an average density value of 0.06 gram/cm³ and the density error percentage obtained was 2.69%. The steel slag composition of 60% in Sample 4 has an average density value of 0.04 gram/cm³ and the density error percentage obtained is 3.95%. The data obtained is in accordance with existing literature, where the greater the steel slag composition used, the density value decreases [25]. The density decreases with increasing variations in the composition of 60% steel slag. This decrease in density is associated with an increase in the volume of pores in the material due to the hydration of the f-CaO components f-CaO (Free Calcium Oxide) and f-MgO (Free Magnesium Oxide) in the steel slag. Increasing the steel slag composition in polyurethane foam causes a hydration reaction which results in volume expansion, thereby reducing the total density and compressive strength of the foam. Higher steel slag composition results in more volume expansion, which increases porosity and reduces the density and compressive strength of the polyurethane foam. The uneven mixing factor during the process of making polyurethane foam can affect the decrease in density.

SEM (Scanning Electron Microscopy) testing was carried out to examine the morphology and pore size of the polyurethane foam. Samples are selected based on the type of foam obtained from the compression test. The purpose of this test is to find out how pore size affects the type of foam produced. Based on Ifa literature in 2018 [26], polyurethane foam that has stiff properties tends to have a smaller pore size, which is known as closed cell. On the other hand, polyurethane foam with flexible properties tends to have larger pore sizes, which are known as open cells. In closed-cell polyurethane foam, the pore structures are isolated from each other, creating greater rigidity and durability. On the other hand, open-cell polyurethane foam has interconnected pores, providing greater flexibility and softness.

In Figure 8, you can see the shape and pore size of Sample 1, Sample 2, Sample 3, and Sample 4. Sample 1 is a foam that has the highest compressive strength value of 0.225 MPa. This foam has oval-shaped and closed pores, with the smallest size being 61.945 μm and the largest size being 376.769 μm , and an average pore size of 135.367 μm . Sample 2 has the second highest

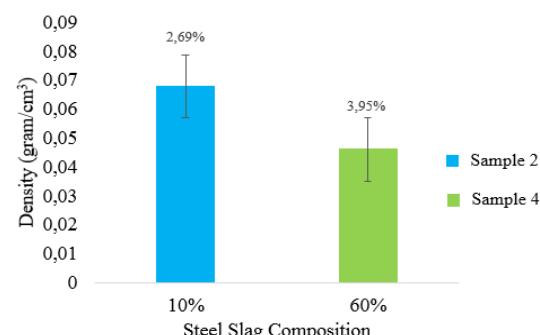


Fig. 7. Graph of %Error Density Values with Steel Slag Composition (a) 10% Sample 2 and (b) 60% Sample 4.

compressive strength value of 0.18 MPa. This foam has oval, slightly round and closed pores, with the smallest size being 49.465 μm and the largest size being 393.816 μm , and an average pore size of 180.880 μm . Sample 4, which is midway between the highest and lowest compressive strength values, has a compressive strength of 0.12 MPa. This foam has round and closed pores, with the smallest size being 86.481 μm and the largest size being 312.838 μm , and an average pore size of 169.222 μm . Sample 3 has the lowest compressive strength value of 0.02 MPa. This foam has irregular round and open pores, with the smallest size being 59.7 μm and the largest size being 413.784 μm , and an average pore size of 173.416 μm . This explanation shows how variations in pore size and shape can affect the compressive strength of polyurethane foam. Closed-pore foam tends to have higher compressive strength due to its denser and more stable structure. In contrast, foam with open and irregular pores, such as in Sample 3, tends to have lower compressive strength. This analysis is important for understanding the relationship between microstructure and mechanical properties of polyurethane foam, which can be used to optimize the material for specific applications.

In Figure 9 to Figure 12, you can see the histogram of each SEM result. In Sample 1, the largest distribution of pores is between 100 and 125 μm in size. Sample 2 shows the largest distribution of pores between 100 and 140 μm in size. For Sample 3, the largest pore distribution is between 100 and 135 μm in size. Meanwhile, Sample 4 shows the largest distribution of pores between 100 and

150 μm in size. This pore size distribution shows variations in the microscopic structure of the resulting polyurethane foam. Larger pore sizes typically correlate with increased flexibility and decreased density, while smaller pore sizes can increase mechanical strength. By understanding this pore size distribution, we can optimize the use of polyurethane foam for specific applications based on desired properties, such as strength and flexibility. These results also show that variations in the composition and manufacturing process of polyurethane foam can significantly influence the microscopic structure and physical properties of the resulting material. It can be seen in Figure 13, for the type of foam shape of each sample, you can see in the picture that on average it has a close cell shape, but there are parts of the foam that show a reticulated foam shape, this makes the foam a mix or mixture of close shapes, cell and reticulated shape. The foam walls have varying thicknesses.

Types of polyurethane foam can be differentiated based on their cell structure, namely close cell, reticulated, and mixed. Polyurethane foam with a closed cell structure has cells that are completely closed and not connected to each other. Its main characteristics are high density, water and air resistance, which is effective because the air is trapped inside the closed cells. In contrast, polyurethane foam with an open cell (reticulated) structure has cells that are connected to each other. This foam is usually lighter and more flexible, allows air and fluid circulation through the foam, and has good compression capabilities and returns to its original shape after the pressure is released.

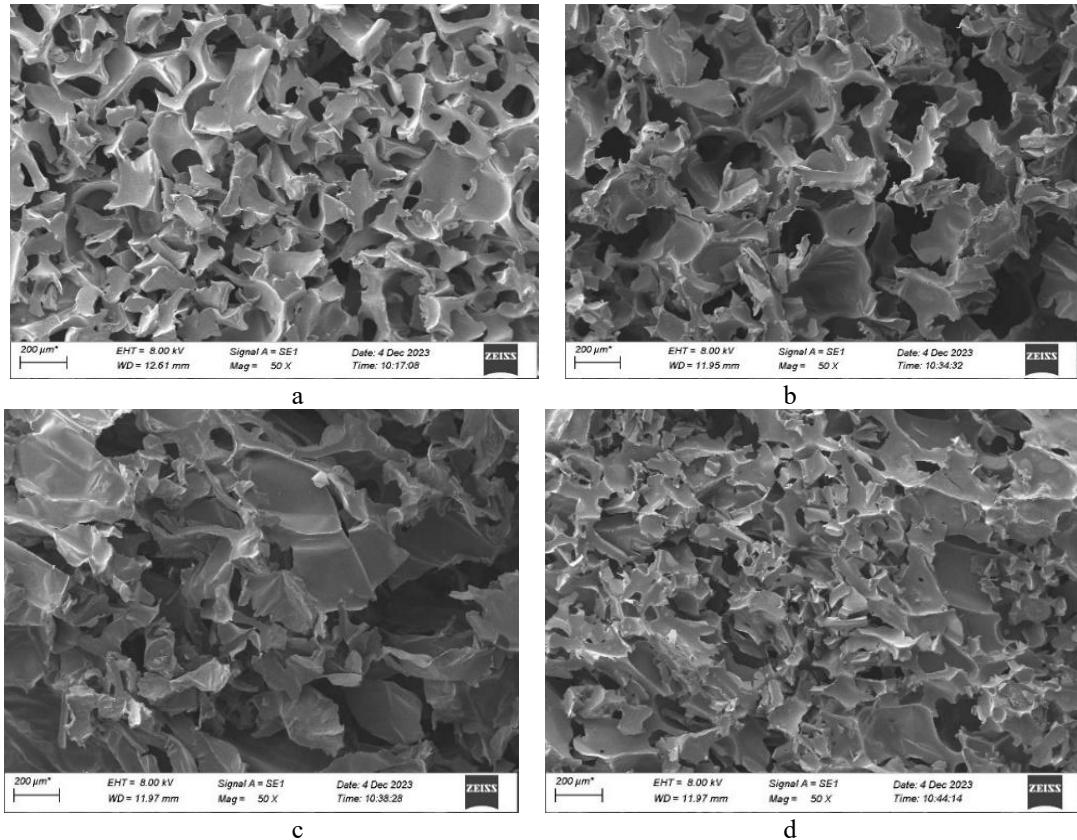


Fig. 8. Results of 50x SEM Magnification of Polyurethane Foam (a) Sample 1 (b) Sample 2 (c) Sample 3 and (d) Sample 4.

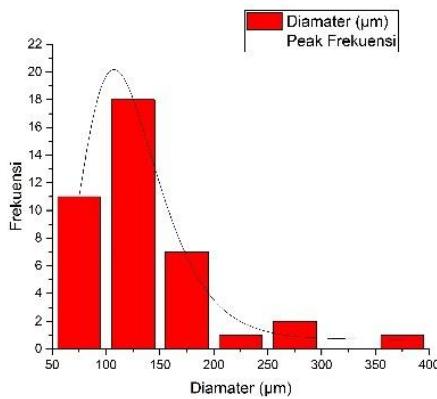


Fig. 9. SEM Histogram of Sample 1

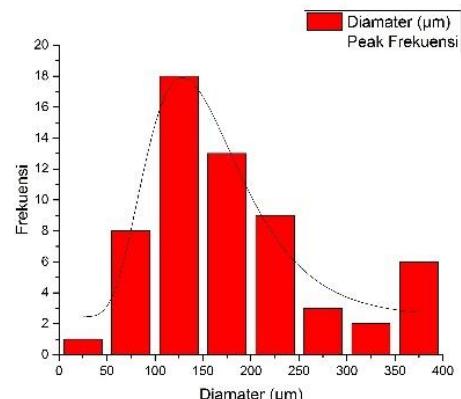


Fig. 10. SEM Histogram of Sample 2

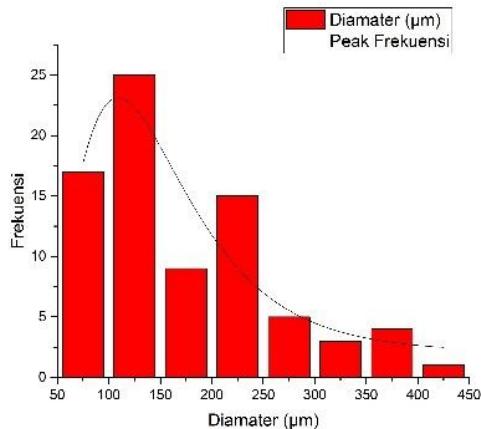


Fig. 11. SEM Histogram of Sample 3

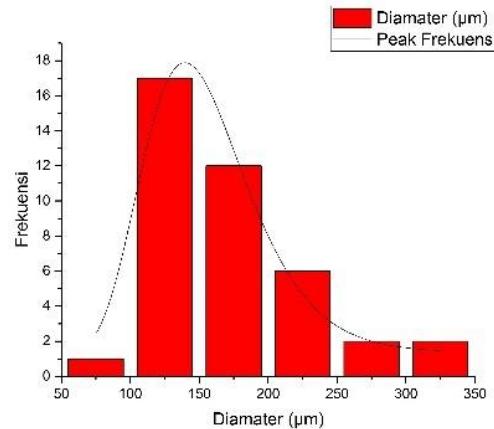
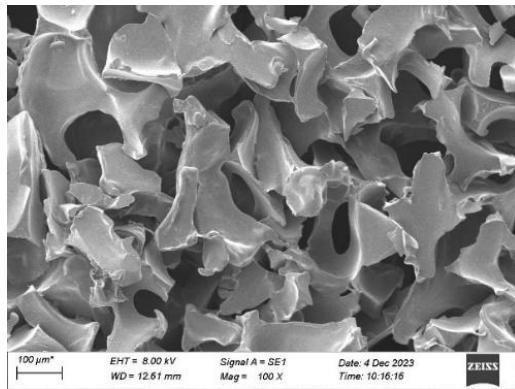
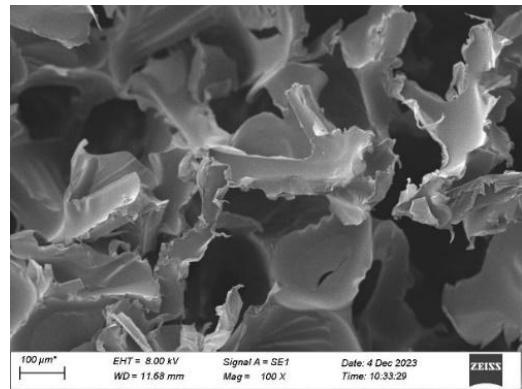


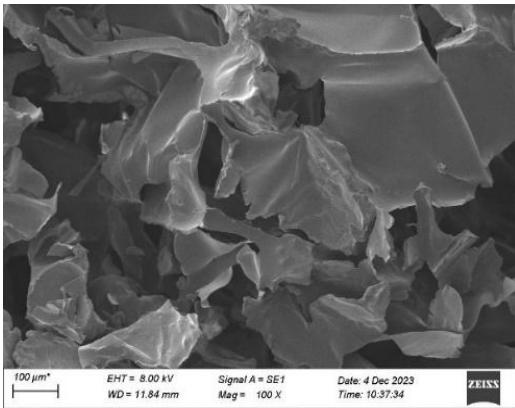
Fig. 12. SEM Histogram of Sample 4



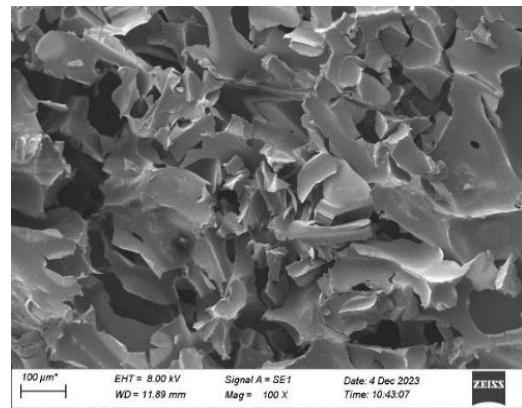
a



b



c



d

Fig. 13. Results of 100x SEM Magnification of Polyurethane Foam (a) Sample 1 (b) Sample 2 (c) Sample 3 and (d) Sample 4.

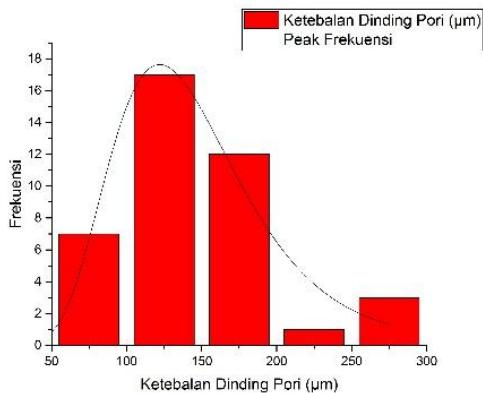


Fig. 14. SEM Histogram of Sample 1.

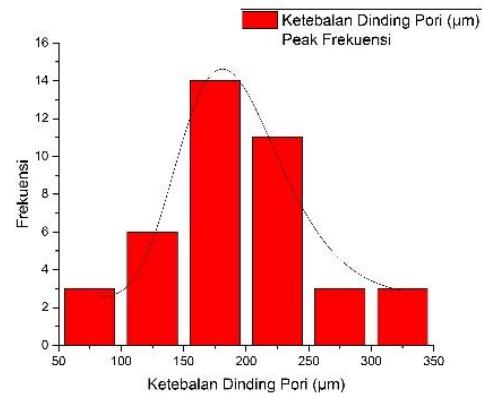


Fig. 15. SEM Histogram of Sample 2.

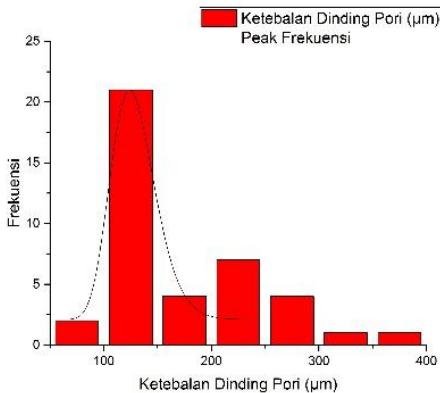


Fig. 16. SEM Histogram of Sample 3.

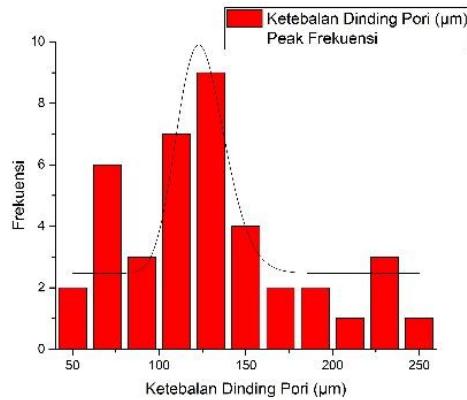


Fig. 17. SEM Histogram of Sample 4.

Polyurethane foam with a mixed structure has a combination of closed cells and open cells, providing the unique properties of both types of cell structures. This mixed structure offers a balance between strength and flexibility. Closed cell sections provide strength and stability, while open cell sections provide ventilation and compressibility. This mixed foam is suitable for applications that require mechanical properties and air or fluid permeability, such as shoe pads and automotive and shoe applications. By understanding these differences, it is possible to determine the right type of foam for a particular application based on the required properties, such as insulation, flexibility, durability, or air and fluid circulation. In Figure 14 to Figure 17, you can see the histogram of the wall thickness of each sample. In Sample 1, the wall thickness is mostly between 110 and 140 μm. For Sample 2, the wall thickness was mostly between 150 and 180 μm. In Sample 3, the wall thickness is mostly between 100 and 125 μm. Meanwhile, in Sample 4, the wall thickness was mostly between 120 and 135 μm.

This explanation shows how variations in wall thickness can affect mechanical properties. Greater wall thickness usually contributes to increased strength and stability of the foam, as thicker walls can withstand greater loads and provide a stiffer structure. For example, Sample 4, with a wall thickness that is mostly in the range of 150 to 180 μm, may have a higher compressive strength than samples with thinner wall thicknesses. On the other hand, thinner wall thicknesses, such as in Sample 3 with wall thicknesses ranging from 100 to 125 μm at most, may provide higher flexibility but with reduced compressive strength. This analysis is important for understanding how

wall thickness affects the overall performance of polyurethane foam and how this material can be optimized for various applications that require a combination of mechanical properties.

Conclusion

The research demonstrates that the glycolysis process using diethylene glycol and zinc acetate is effective in recycling PET and HDPE waste into polyurethane foam materials. The addition of steel slag from steel smelting waste introduces potential to improve mechanical properties, although an increase in steel slag composition results in greater CO₂ formation, which negatively impacts foam strength and density. Among the produced foams, Sample 1 exhibited the best mechanical properties, with the highest compressive strength, Young's modulus, and yield strength. The density of Sample 1 was also the highest, with a minimal error percentage, suggesting its superior structural integrity.

On the other hand, Sample 3, with the lowest steel slag composition, displayed the weakest mechanical properties, showing the lowest compressive strength, Young's modulus, and yield strength, alongside the smallest density. Pore size analysis further reinforced these findings, with Sample 1 having the most optimized pore structure for strength, while Sample 3 had larger, less favorable pore sizes. These results indicate that while steel slag can enhance foam properties, controlling its composition is key to maintaining desirable material performance.

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Declarations

Conflict of interest. The authors declare that they have no known conflicts of interest.

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Виготовлення поліуретанового шлакового композиту з використанням поліолу, отриманого з поліетиленових пластикових відходів

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Метою даного дослідження є розробка поліуретанової піни з використанням перероблених пластикових пляшок PET (поліетилентерефталат) та HDPE (поліетилен високої щільності) як замінників поліолу. Відпрацьовані пляшки PET переробляли методом гліколізу з утворенням ВНЕТ (біс(гідроксіетил)терефталату), який використовувався як замінник поліолу у виробництві поліуретанової піни. Піну синтезували шляхом реакції поліолу з метилендіфенілдіізоціанатом (MDI), варіюючи вміст дистильованої води як газоутворювача, силікону як поверхнево-активної речовини та сталевого шлаку (10 %, 10 %, 10 % та 60 %) для підвищення механічних властивостей. Було випробувано чотири зразки поліуретанової піни, в результаті чого отримано жорсткі, гнучкі та напівжорсткі піни залежно від рецептури. Зразок 1 продемонстрував межу міцності при стиску 0,225 МПа, модуль Юнга 0,0139 МПа, межу текучості 0,174 МПа та густину 0,11 г/см³. Зразок 2 мав межу міцності при стиску 0,18 МПа, модуль Юнга 0,0109 МПа, межу текучості 0,117 МПа та густину 0,06 г/см³. Зразок 3 характеризувався найменшими значеннями межі міцності при стиску (0,02 МПа), модуля Юнга (0,00079 МПа), межі текучості (0,0092 МПа) та густини (0,09 г/см³). Для зразка 4 межа міцності при стиску становила 0,12 МПа, модуль Юнга – 0,0116 МПа, межа текучості – 0,0901 МПа, густина – 0,04 г/см³. Найвищі механічні характеристики продемонстрував зразок 1, тоді як найнижчі – зразок 3. Отримані результати свідчать, що поліуретанова піна з оптимальними значеннями межі міцності при стиску, модуля Юнга, межі текучості, густини та гнучкості може бути отримана відповідно до вимог стандарту SNI (Standar Nasional Indonesia) 0111-2009.

Ключові слова: композит, поліол, поліуретанова піна, переробка, сталевий шлак.