

Research Article

Utilization of Cassava Processing Liquid Waste as Raw Material for Making Biodegradable Plastics with the Addition of Glycerol Plasticizer

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Abstract: This study aimed to utilize cassava processing liquid waste into biodegradable plastic with glycerol as a plasticizer. This experimental study varies the amount of glycerol: (0, 1, 2, 3, 4, and 5) mL. The resulting biodegradable plastics were analyzed for physical properties consist of (water content and degree of swelling), mechanical properties consist of (tensile strength, elongation, elasticity), biodegradation, and structural properties. Fourier Transform Infra-Red and x-ray diffraction were employed for the studies. The results showed that the more glycerol added, the greater the percentage of water content and the degree of swelling were obtained. The optimum tensile of the biodegradable plastic was reached with 3 mL of glycerol, giving the strength of 27.49 N/mm², elongation 0.107 N/mm², elasticity 4.804 MPa. Biodegradable plastic with the addition of 5 mL of glycerol degraded up to 60.777%. The FTIR spectra showed almost the same peaks between plastics without the addition of glycerol and glycerol. Meanwhile, XRD data shows that the degree of crystallinity of plastic without glycerol is higher than that of plastic with the addition of glycerol.

Keywords: Biodegradable plastic, cassava processing wastewater, plasticizer

Introduction

Cassava (*Manihot esculenta*) is a widely available plant in Indonesia, especially in West Sumatra. The people of West Sumatra use the plant because it contains chemicals that are useful for the body. The chemical content of 100 grams of cassava meat is 1 gram of protein, 15 grams of calories, 36.8 grams of carbohydrates, and 0.1 grams of fat [1]. The community processes cassava tubers into various types of food, such as Karak Kaling, Chips, and Tapai. At the time of processing, it will produce liquid waste that can pollute the environment. Cassava processing liquid waste can be used because it still contains starch, which can be developed as a raw material for biodegradable plastics [2]. Biodegradable plastics are environmentally friendly plastics that are readily decomposed by certain microorganisms [3]. Biodegradable plastic has an essential role in reducing the impact of pollution due to the use of plastic at this time. The plastics that people use today are synthetic.

Synthetic plastics are petrochemical polymer plastics derived from depleting and non-renewable petroleum [4]. Synthetic plastic is difficult to degrade, causing the accumulation of plastic waste, disturbing soil fertility, and damaging the surrounding environment [1]. In addition, this plastic is not safe to use as food packaging. Monomers from synthetic plastics can move into food and then into the human body that consumes it. The chemicals from these plastics are insoluble in water, so they cannot be removed from the body. The buildup of these chemicals is harmful to the body and can lead to cancer [5]. The alternatives are made to reduce this impact. One of them is developing environmentally-friendly plastics such as biodegradable plastics.

Biodegradable plastic is a plastic that can be renewed because it comes from nature. Biodegradable plastics derived from nature are quickly degraded and decomposed by microorganisms. Biodegradable plastic is degraded to form H₂O and CO₂ compounds [3]. In addition, biodegradable plastics can inhibit

the diffusion of oxygen and water vapor into the coated material, inhibit microbial spoilage, and are safe for consumption [6]. One of the natural materials used to manufacture biodegradable plastic is cassava because it has a high starch content. However, biodegradable plastics formed from starch are relatively stiff, so it is necessary to add a plasticizer to make the plastic more elastic [7].

Plasticizers that can manufacture biodegradable plastics are glycerol, sorbitol, chitosan, and polyethylene glycol. The most commonly used plasticizer is glycerol. Glycerol can be used as a plasticizer because it is hydrophilic and easily soluble in water. The hydroxyl group in glycerol causes bonds between starch and glycerol as a substitute for hydrogen bonds between starch polymers during the formation of biodegradable plastic biopolymers. Each addition of glycerol will affect the shape of the biodegradable plastic [8].

Previous research on the effect of storage time on mechanical properties obtained a perfect plastic, namely at 12 grams of starch with the addition of 4 ml of glycerol, the tensile strength was 0.2122 kgf/mm² and would be degraded in 2 weeks [1]. The type of plasticizer influences the physical and mechanical properties of biodegradable plastics. A suitable plasticizer for the manufacture of biodegradable plastic is glycerol. In the preliminary test, biodegradable plastic from cassava starch with glycerol plasticizer has obtained biodegradable plastic sheets. On this plastic sheet, there are air bubbles, stiff and easy to tear. It is caused by the addition of plasticizers in the manufacture of plastic that is not appropriate. For this reason, it is necessary to look at the addition of a suitable plasticizer to obtain biodegradable plastic following the standard.

Based on these problems, this research utilized cassava processing liquid waste as raw material for making biodegradable plastics with the addition of a glycerol plasticizer. This research using cassava processing wastewater is expected to obtain good physical and mechanical properties of biodegradable plastic with glycerol as a plasticizer.

Materials and Methods

Tools and Materials

The tools used in this study were 100 mL beaker, blender, oven, sieve, knife, analytical balance, measuring cup, tripod, gauze, spirit, stirring rod, biodegradable plastic mold, and the instruments used were tensile strength, Fourier Transform Infra-Red (FTIR), X-ray Diffraction (XRD). The materials used in this study were cassava processing wastewater, distilled water, acetic acid (CH₃COOH) (PT. Bratachem), and pure glycerol (PT. Bratachem).

Type of Research

This experimental study varies the amount of glycerol: (0, 1, 2, 3, 4, and 5) mL. The resulting biodegradable plastics were analyzed for physical properties consist of (water content and degree of swelling), mechanical properties consist of (tensile strength, elongation, elasticity), biodegradation, and structural properties. Fourier Transform Infra-Red and x-ray diffraction were employed for the studies.

Manufacture of Dry Starch from Cassava Processing Waste

As much as 5 L of cassava processing liquid waste can stand until it separates and a precipitate is formed. The precipitate is then filtered to separate it from the filtrate. Furthermore, the precipitate was baked at a temperature of 70 °C for 1 hour, obtained dry starch from cassava processing wastewater.

Biodegradable Plastic Manufacturing

The dry starch was weighed as much as 12 grams and put into a beaker. Then add 25 mL of distilled water, 3 mL of acetic acid, and X mL of glycerol (with variations of 0, 1, 3, 4, and 5 mL), which function as plasticizers plastic becomes elastic and heated while stirring until homogeneous. The solution is heated at 70-80 °C while stirring until it turns into a gel. The mixture that has formed a gel is lifted and printed on a prepared glass mold. Then the mixture is dried at room temperature until a stable gel is formed (biodegradable plastic film).

Biodegradable Plastic Characterization

The resulting biodegradable plastics were analyzed for physical properties consist of (water content and degree of swelling), mechanical properties consist of (tensile strength, elongation, elasticity), biodegradation, and structural properties. Fourier Transform Infra-Red and x-ray diffraction were employed for the studies [9] [10].

FTIR testing will show how the absorption of polymer groups in the sample will be known so that the bonding groups contained in the sample will be known. The bonding group in the sample can be known based on the graph that appears on the computer screen as a device connected to FTIR (Amalia, 2012). Analysis using spectrophotometry can produce a typical absorption spectrum for each functional group in the sample. FTIR analysis on biodegradable plastic aims to determine the mixing process by looking at the functional groups contained in the sample. The sample in the form of film is placed into

the set holder; then, the spectrum is searched. The result can be a diffractogram of the relationship between wavenumber and intensity. The FTIR spectrum can be recorded at room temperature [11].

Crystallinity is an essential property of polymers that shows the bonds between molecular chains to produce a more regular molecular arrangement. High crystallinity properties cause high stress and stiffness. The polymer chain structures have different crystallinities. The crystallinity of the polymer is influenced by the type of chain structure and the type of bond. The degree of crystallinity can be studied through thin films using X-ray diffraction [12].

Results and Discussions

Dried Starch

The making of biodegradable plastic involves a deposition process that separates water from sediment in cassava processing liquid waste. The starch precipitate is heated to reduce the water content contained in the starch. This reduction in water content is made so that the growth of microorganisms can be inhibited so that starch can last longer. Starch is a polysaccharide consisting of amylose and amylopectin. Starch from cassava can be used as raw material for making biodegradable plastics because it has plastic-like characteristics. Plastics from starch are biodegradable and abundant in nature. Dry starch is obtained by precipitation process in powder, and white color can be seen in (Figure 1).



Figure 1. Dried Starch

Biodegradable plastic

Biodegradable plastic from starch is stiff and brittle, so the plastic breaks easily. The properties of this biodegradable plastic can be improved by adding a plasticizer. One of the plasticizers used is glycerol. Glycerol can reduce intermolecular forces on polymer chains, thereby increasing the flexibility of biodegradable plastics, widening intermolecular distances, and reducing hydrogen bonds in polymer chains [13]. Each addition of glycerol affects the resulting biodegradable plastic. The manufacture of biodegradable plastics from cassava starch involves heating to allow the gelatinization process to occur. The gelatinization process destroys intermolecular hydrogen bonds. Hydrogen bonds in starch play a role in maintaining the structural integrity of the granules. The broken hydrogen bonds cause water to enter the granules, causing swelling of the starch granules. The previously free water outside the starch granules becomes trapped and cannot move freely again after gelatinization. The gel formed after heating was dried at room temperature to shrink due to the release of water to form a stable film. Biodegradable plastic without the addition of glycerol and glycerol can be seen in (Figure 2).

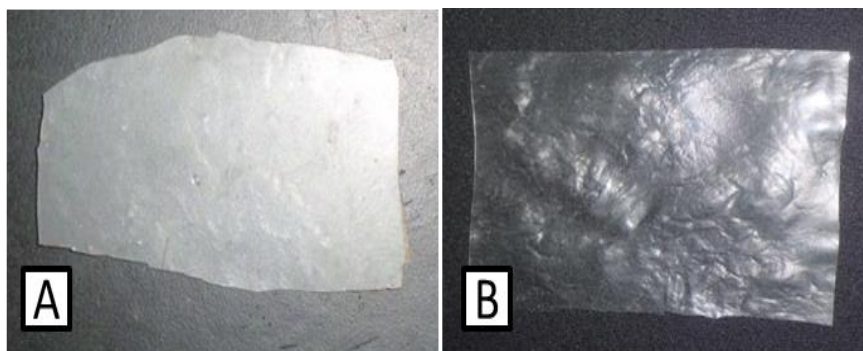


Figure 2. Biodegradable plastic (A) Without the addition of glycerol (B) The addition of glycerol.

Characterization of the Physical Properties of Biodegradable Plastics

The obtained biodegradable plastic was tested for physical properties. The physical properties of biodegradable plastic are analyzed for water content, and the degree of swelling can be seen in (Table 1).

Table 1. Biodegradable plastic physical properties data

Addition of Glycerol (mL)	% Water content	% degree of swelling
0	11,500	25,452
1	13,922	36,546
2	15,717	48,464
3	18,045	52,154
4	18,906	66,492
5	20,639	68,139

Water content is a parameter of physical properties test to determine how much water is contained in biodegradable plastic. The water content test is the amount of water content in biodegradable plastic compared to the dry weight of the plastic. The effect of adding glycerol as a plasticizer in biodegradable plastics on the percentage of water contained in the plastic can be seen in (Figure 3).

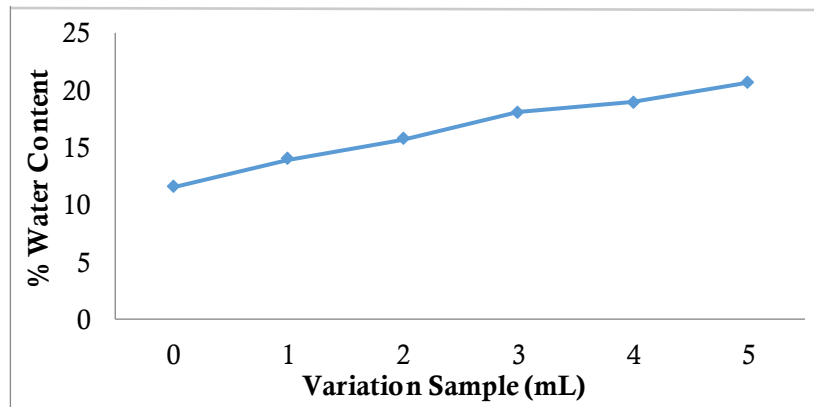


Figure 3. The effect of adding glycerol to the water content of biodegradable plastics

Based on (Figure 3), the percentage of water content increases with the addition of glycerol. The addition of glycerol can make the plastic loose-fitting because there are empty cavities in the plastic. Glycerol increases the water content of the resulting biodegradable plastic. Glycerol has a hydroxyl group that can form hydrogen bonds so that during the drying process, water is difficult to evaporate. It causes the water content to increase [14].

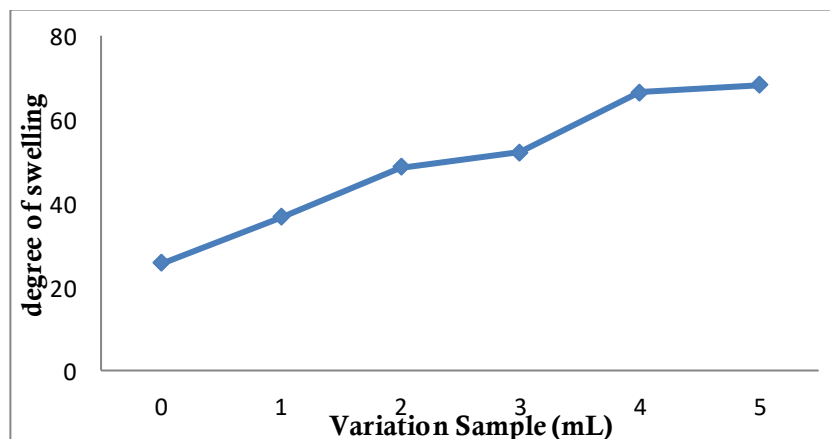


Figure 4. The effect of adding glycerol to the % degree of swelling of biodegradable plastics

The degree of swelling is one of the parameters of biodegradable plastic's physical properties, which states how much the sample can swell when interacting with the solvent (water) over a certain period. The degree of swelling helps estimates the shelf life of the product to be stored. Based on the research that has been done, the percentage of swelling degrees obtained by biodegradable plastic without the addition of glycerol and variations of glycerol 1, 2, 3, 4, and 5 mL, respectively, are 25.452%, 36.546%, 48.464%, 52.154%, 66.492%, and 68.139%. The degree of swelling percentage can be seen in (Figure 4).

The results obtained from the determination of the degree of swelling indicate that the volume of glycerol increases the swelling percentage. The addition of glycerol can increase the hydrophilic properties of the resulting plastic so that the more glycerol is added; the plastic absorbs much water. The amount of free space (free volume) increases along with glycerol. It will increase the gap to be occupied by water molecules [15].

Biodegradable Plastic Mechanical Properties

The obtained biodegradable plastic was tested for mechanical properties. The mechanical properties of biodegradable plastic are analyzed for tensile strength, elongation, and elasticity can be seen in (Table 2).

Table 2. Mechanical properties of biodegradable plastic

Addition of Glycerol (mL)	Stress (N/mm ²)	Strain (N/mm ²)	Young's modulus (MPa)
1	20,45	0,031	2,749
2	23,81	0,066	4,134
3	27,49	0,107	4,804
4	21,44	0,078	2,727
5	18,46	0,069	1,572

Tensile strength testing is one of the essential parameters in the manufacture of the resulting plastic. The higher the tensile strength, the better the quality of the glycerol biodegradable plastic produced. The effect of adding glycerol to biodegradable plastics can be seen in (Figure 5).

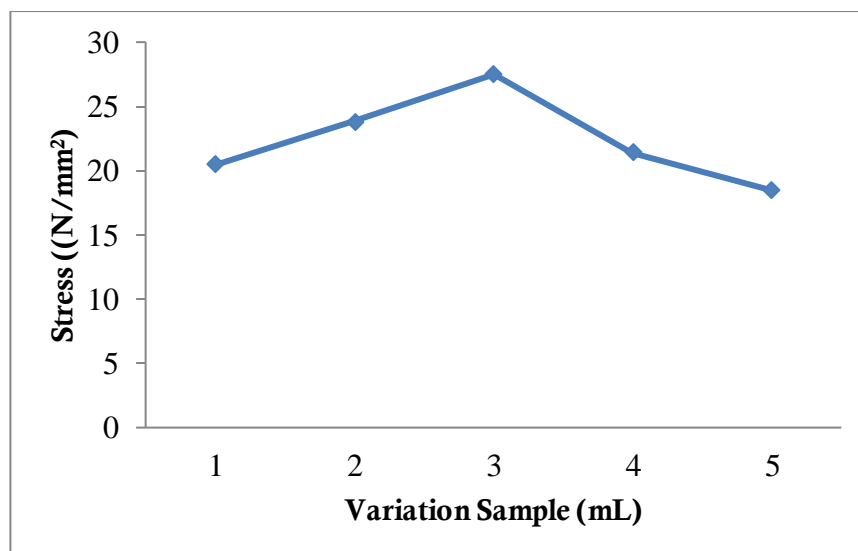


Figure 5. The effect of adding glycerol to the tensile strength of biodegradable plastics

Based on (Figure 5), the tensile strength of plastic increases with the addition of glycerol. The most excellent tensile strength value was owned by biodegradable plastic with the addition of 3 mL glycerol. However, the tensile strength value of glycerol plastic decreased with the addition of 4 mL glycerol. The decrease in tensile strength is related to the space because the bonds between starches are broken by glycerol. The intermolecular bonds in the plastic film are getting weaker. The addition of glycerol causes a decrease in the tensile strength value. It is due to the reduced intermolecular interactions that insert and

remove hydrogen bonds between starch molecules. Biodegradable plastic that has high tensile strength will be able to protect the packaged product [16].

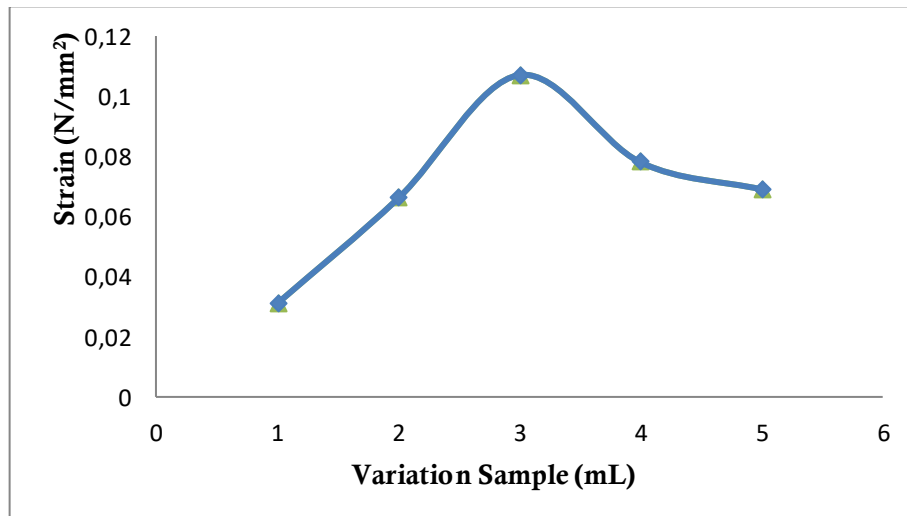


Figure 6. The effect of adding glycerol to the elongation of biodegradable plastics

Elongation is a mechanical property that shows the maximum plastic length change when obtaining a tensile force until the plastic breaks. Based on (Figure 6), the effect of glycerol on the elongation ability of the plastic continues to increase in the amount of glycerol added. The addition of glycerol is directly proportional to the elongation percentage. The significantly the addition of glycerol makes the greater the value of the percentage of elongation.

Glycerol can increase the percentage of elongation so that the plastic can become elastic and reduce the brittleness of plastic. Glycerol can make plastics elastic by disrupting hydrogen bonds between adjacent polymer molecules so that the intermolecular attractions of polymer chains are reduced. However, the addition of 4 mL of glycerol decreased the percentage of elongation [17]. Adding more glycerol will affect the more negligible cohesion bonds between polymers, and the resulting plastic becomes softer so that the resulting biodegradable plastic is quickly broken [18].

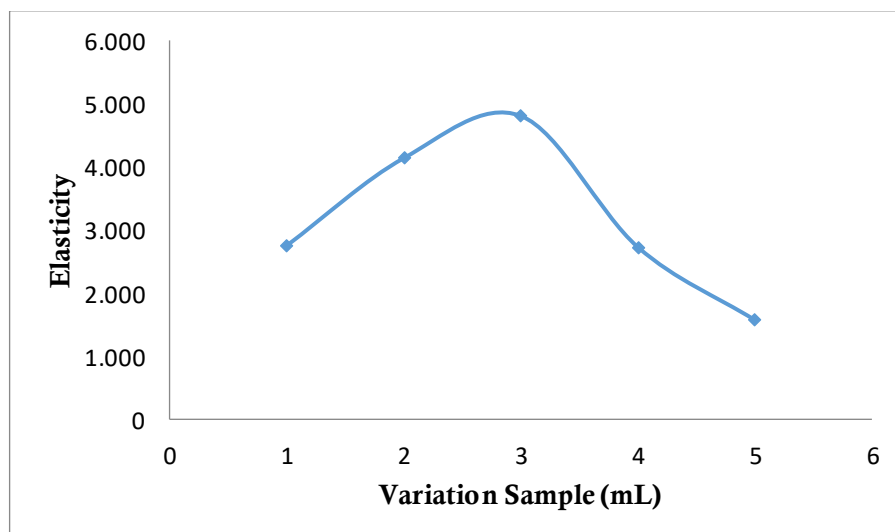


Figure 7. The effect of adding glycerol to the elasticity of biodegradable plastics

Elasticity is a measure of the strength of the plastic produced. Based on (Figure 7), it can be seen that the plastic with the addition of glycerol reaches its maximum elasticity at the addition of 3 mL, which is 4.804 MPa. According to [19], the more significant the concentration of plasticizers, the more elastic the material is; on the contrary, the smaller the degree of stiffness or Young's modulus.

Biodegradation of Biodegradable Plastic

The biodegradability test aims to determine the ability of plastic degradation obtained in a specific time interval. The degradation test in this study was carried out by utilizing microorganisms found in the environment. Microorganisms available in the environment will convert plastics into natural substances, such as water, carbon dioxide, and compost (unnecessary additives). The biodegradation test was determined by examining the effect of burial time on the percent loss of sample weight and the sample's surface before and after burial in the soil. The biodegradation test of biodegradable plastic is characterized by damage to the plastic sheet so that it loses weight which indicates that the polymer chain bonds are breaking. The breaking of chain bonds in polymers is caused by microbial decomposers, soil moisture, soil pH, soil temperature, and other physical and chemical factors present in the soil [20]. The plastic that has been obtained has been tested for biodegradation, which can be seen in (Table 3).

Table 3. Biodegradation of Biodegradable Plastic

Sample	0 Days	3 Days	6 Days	9 Days	12 Days	15 Days
0	-	14,796	17,683	21,384	22,734	29,205
1	-	19,156	22,321	25,839	32,109	35,379
2	-	24,101	26,210	31,562	39,419	45,201
3	-	32,046	38,658	41,348	46,276	48,631
4	-	39,951	43,738	49,869	51,058	52,572
5	-	44,444	51,869	53,820	55,502	60,777

Biodegradable plastic with the addition of 5 mL of glycerol degraded up to 60.777% for 15 days. The more glycerol added, the easier the plastic will degrade. Degraded plastic is characterized by plastic damage after burial. Plastic damage after burial can be seen in (Figure 8)

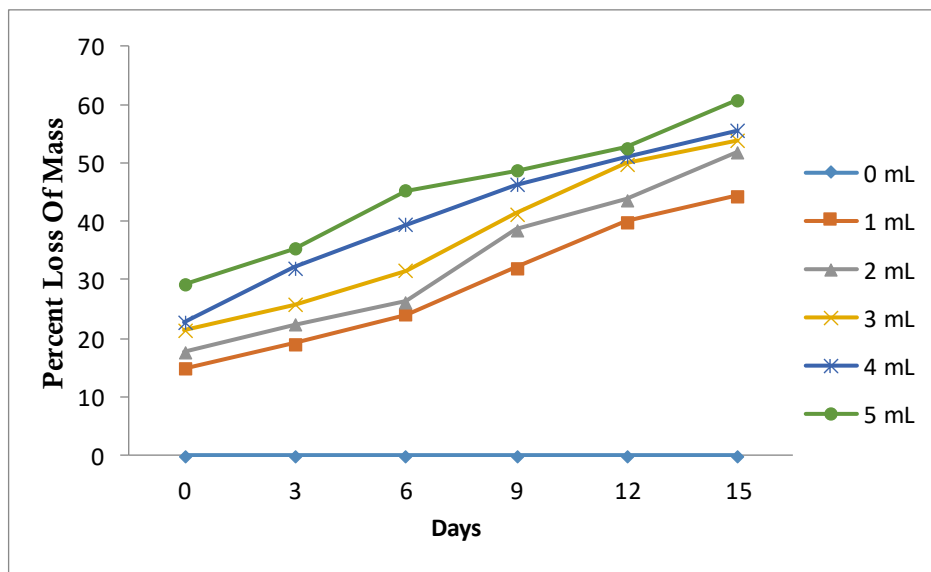


Figure 8. The effect of adding glycerol to the biodegradation of biodegradable plastics

Biodegradation analysis is also carried out visually. Figure 8 shows the appearance of plastic on day 0, which looks intact and has not been damaged. The burial on the 15th day of plastic underwent a slight change marked by the surface of plastic with holes and a change in color to red spots on the plastic. Plastic will continue to experience structural changes, and the pores will get more significant due to the microbes in the soil. According to [15], the biodegradability of plastics increases with the amount of glycerol added. It is related to the ability of plastic to absorb water; namely, the more water content of a material, the easier it is for the material to be degraded. Water is a medium for most microbes and bacteria. Apart from the nature of the constituent components that are easily degraded naturally, the assistance of bacteria and decomposers such as earthworms can accelerate the rate of plastic degradation.

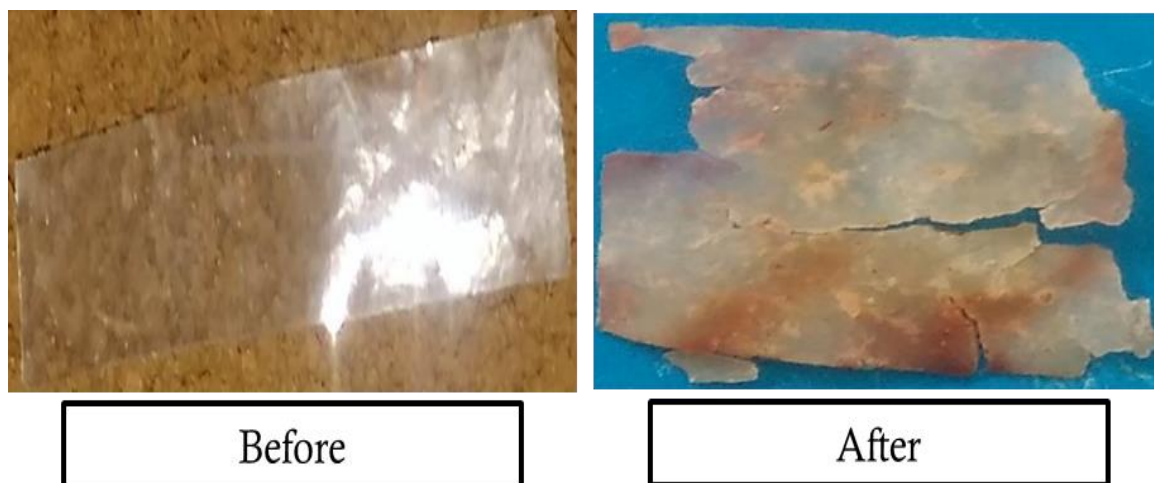


Figure 9. The difference between biodegradable plastics before and after being degraded in the soil

Biodegradable plastics have essential ingredients under certain conditions and will experience changes in chemical structure that microorganisms can influence at a specific time. Biodegradable plastics made from starch can be degraded by *Pseudomonas* and *Bacillus* bacteria, breaking polymer chains into monomers [21]. Plastic is degraded due to the presence of glycerol. Glycerol and starch have OH groups that can initiate hydrolysis reactions after absorbing water from the soil. Then, the starch polymer will be degraded due to damage or decreased quality due to breaking the chain bonds in the polymer [22].

Characteristics of Biodegradable Plastic Molecular Structure

The obtained biodegradable plastics were characterized by their molecular structure using FTIR and XRD. The FTIR instrument aims to determine the functional groups contained in the obtained biodegradable plastic. The functional groups and wavenumbers of plastic without the addition of glycerol with 3 mL of glycerol can be seen in (Table 4).

Table 4. The peak of the wavenumber in each functional group

The Peak (cm ⁻¹)	Plastic without the addition of glycerol	Plastic with the addition of 3 mL of glycerol	Absorption area (cm ⁻¹)
O-H	3293,11 cm ⁻¹	3303,71 cm ⁻¹	3650-3200
C-H	2924,77 cm ⁻¹	2930,24 cm ⁻¹	3000-2840
C=C	1643,28 cm ⁻¹	1645,41 cm ⁻¹	1820-1640
C-O	1005,42	928,98 cm ⁻¹	1300-900
C-O-C	1139,02 cm ⁻¹	1014,49 cm ⁻¹	1200-900

Based on (Figure 10), there is a widening peak and a decrease in the intensity of the group being passed. The decreasing number of OH groups of glycerol in the plastic is due to the insertion of glycerol molecules in the intermolecular hydrogen bonds of starch [15]. The O-H alcohol group on biodegradable plastic without the addition of glycerol and biodegradable plastic with 3 mL of glycerol were found at wavenumbers 3293.11 cm⁻¹ and 3303.71 cm⁻¹. Absorption at wave number 1139.02 cm⁻¹ on biodegradable plastic without the addition of 3 ml glycerol and 1014.49 cm⁻¹ on biodegradable plastic with the addition of glycerol indicated the presence of a pyranose ring and glycosidic bonds. Based on the spectral data in Figure 15, it shows that without the addition of glycerol in the manufacture of biodegradable plastics with the addition of glycerol, no new functional groups were found, only that the peak intensity of plastic with the addition of glycerol was weaker than that of plastic without the addition of glycerol. It shows that making biodegradable plastic is a physical mixing process in hydrogen bonds between chains. The plastic produced is a physical blending process because no new functional groups exist. Only chemical interactions occur because there is only a shift in wavenumber [10].

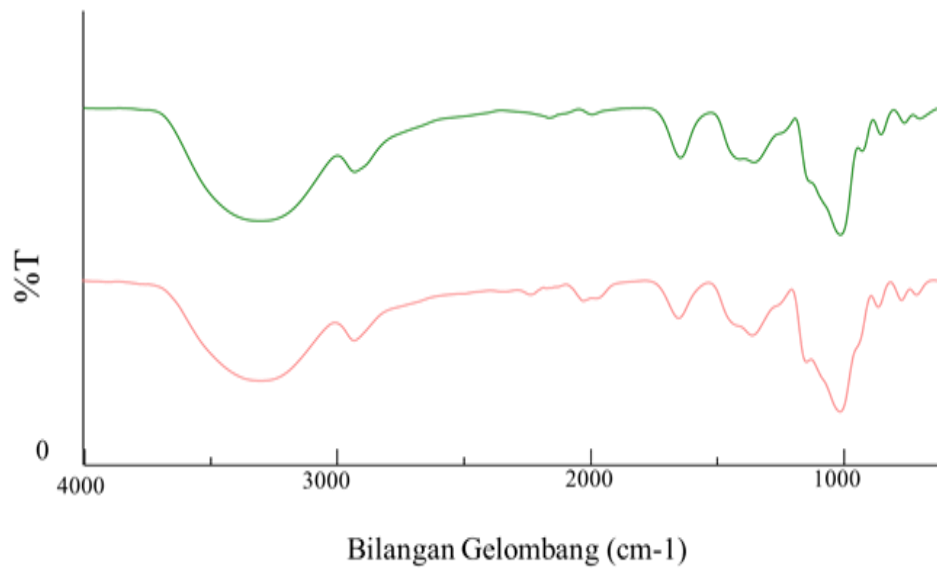


Figure 10. Green line no addition of glycerol and red line addition of 3 mL of glycerol

The obtained biodegradable plastics were characterized using XRD to determine whether the plastic is crystalline or amorphous. The degree of crystallinity of biodegradable plastic without glycerol and the addition of 3 mL of glycerol can be seen in (Table 5).

Table 5. Degree of crystallinity of biodegradable plastic

Sample	Total Weight	Crystal weight	Amorphous Weight	Crystallinity
without the addition of glycerol	2287,521	2226,502	61,01932	0,973
addition of 3 mL of glycerol	336,17	219,455	116,715	0,653

Based on calculations, the degree of crystallinity without glycerol is 0.973. In contrast, plastic with the addition of 3 mL of glycerol has a degree of crystallinity of 0.653. It shows that the degree of crystallinity of the plastic without glycerol is higher than the plastic with glycerol, causing the plastic without glycerol to be stiffer. The higher the degree of crystallinity, the elasticity of a material will decrease.

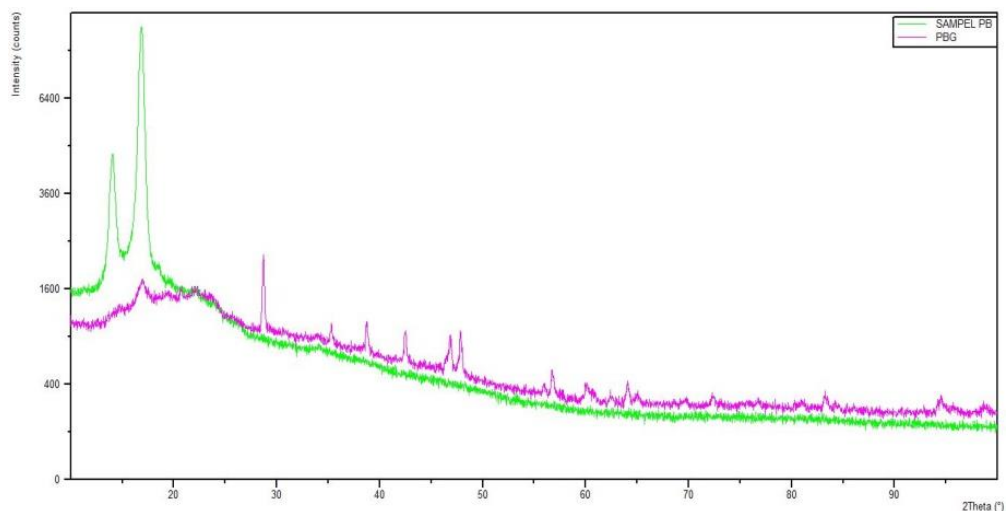


Figure 11. Diffractogram Biodegradable plastic without the addition of glycerol with the addition of glycerol

The analysis of the degree of crystallinity of biodegradable plastics aims to determine the structure of biodegradable plastics, including crystalline or amorphous plastics. Polymers can have crystalline regions that randomly mix with amorphous regions. X-ray diffractograms of crystalline polymers produce sharp peaks, whereas amorphous polymers tend to produce broad peaks. This information can be obtained from an X-ray diffractogram [23]. A plastic diffractogram of cassava without glycerol with the addition of glycerol can be seen in (Figure 11). The degree of crystallinity affects the degradation power. Amorphous plastics are easily degraded. Decomposing microorganisms first attack the amorphous part of the plastic in the soil. The enzyme biodegrades the amorphous part more than the crystalline part. The amorphous part contains more functional groups that can act as a substrate than the crystalline part [23].

Table 6. Biodegradable Plastic Particle Size

Sample	2 θ	FWHM Left	Crystal size
without the addition of glycerol	16.9194	0.2303	27, 31321
addition of 3 mL of glycerol	17.0240	0.3582	37,88207

The particle size of the biodegradable plastic without glycerol and the addition of 3 mL of glycerol can be seen in (Table 6). The particle size of biodegradable plastic with 3 mL of glycerol resulted in larger grain size than without glycerol. The biodegradable plastic with the addition of 3 mL of glycerol has been substituted for glycerol plasticizer, affecting the particle size of biodegradable plastic.

Conclusion

The addition of glycerol affects the physical properties, mechanical properties, and plastic structure. Physical properties which are, water content and degree of swelling, increase with the addition of glycerol. Mechanical properties are elasticity, increase with glycerol, and maximum at 3 mL of glycerol. Biodegradable plastic with the addition of 3 mL of glycerol has a tensile strength of 27.49 N/mm², an elongation of 0.107 N/mm², and an elasticity of 4.804 MPa. The biodegradation test shows that the more glycerol added, the easier the plastic will degrade. Biodegradable plastic with the addition of 5 mL of glycerol degraded up to 60.777%. The FTIR spectra showed almost the same peaks between plastics without the addition of glycerol and glycerol. Meanwhile, XRD data shows that the degree of crystallinity of plastic without glycerol is higher than that of plastic with the addition of glycerol.

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