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Bioplastic Manufacturing from Durian Rind Cellulose Using the Phase Inversion Method

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ABSTRACT

Plastic waste in Indonesia in 2020 reached around 5.66 million tons, or 17.1% of the country's total waste based on Data from the Ministry of Environment and Forestry of the Republic of Indonesia (2020). Using bioplastics is one solution to this problem. Bioplastics are plastics made from biodegradable materials such as cellulose. Durian rind contains high levels of cellulose (50 - 60%) and lignin (5%). Durian rind cellulose can be extracted through delignification. Phase inversion is a process in which a polymer is changed in a controlled manner from a liquid to a solid state. Phase inversion method is used to make bioplastics from durian rind cellulose with the addition of chitosan and glycerol to test for the effect in mechanical test properties of bioplastic (tensile strength, elongation, swelling and biodegradation tests). Bioplastics are made with variations chitosan levels of 8%, 10%, 12%, 14% and 16% and variations glycerol levels of 1%, 2%, 3%, 4%, 5%. The best bioplastic from durian rind cellulose is the variation of 16% chitosan and 1% glycerol. The bioplastic has a tensile strength value of 1.1603 Mpa, biodegradation of 98.99% in 12 days and swelling (water absorption capacity) of 25.11% and elongation of 12.20. %.

Keywords: Corruption crimes, return of state financial losses

Introduction

According to Mulyadi (2019), Data from the Ministry of Environment and Forestry of the Republic of Indonesia 2020 shows that plastic waste in Indonesia in 2020 reached around 5.66 million tons, or 17.1% of the country's total waste. Using bioplastics is one solution to this problem. Bioplastics are plastics that use a mixture of natural materials such as cellulose and starch. Bioplastics can be made from several materials, one of which is cellulose. Cellulose is a polymer that can be renewed, biocompostable, and easily degraded. With high biodegradability and the abundance of cellulose in nature, bioplastics can be made.

Durian fruit (*Durio zibethinus Murr*) is a fruit that is popular with many people. However, according to Nugraheni's research (2018), the edible part of the fruit (percentage of fruit flesh weight) was only 20.52%, which means that there was around 79.08% of the fruit that was not used for eating, such as durian rind and seeds, which were estimated to be produced. around 556,360 tons of durian rind waste every year. Proportionally, durian rind contains high levels of cellulose (50 - 60%) and lignin (5%). Higher cellulose content has many benefits.

Durian rind cellulose has been used in several studies. One example is research conducted by Ariyani (2012), namely the use of durian rind as raw material for decorative paper. Another research by Mashuni (2021) created bioplastic from chitosan with variations of durian rind cellulose as antibacterial food packaging with a mixture of 12% chitosan, 2% glycerol, and 6% cellulose. This bioplastic has a tensile value of 5.16, elongation of 3.01, thickness of 0.23, and biodegradation value of 17.94 percent.

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Delignification is carried out using a NaOH solution because this solution can damage the lignin structure in the crystalline and amorphous parts and separate some of the hemicellulose. Hemicellulose has an amorphous structure so the use of NaOH can remove lignin while extracting hemicellulose. The OH ions from NaOH will break the bonds of the basic structure of lignin, while the Na+ ions will bond with lignin to form sodium phenolate. This phenolic salt is easily soluble (Zely, 2014).

Phase inversion is a process in which a polymer is changed in a controlled manner from a liquid to a solid state. The solidification process is often initiated by the transition of one liquid state into two liquids (liquid-liquid demixing). At a certain stage during demixing, one of the liquid phases (the high polymer concentration phase) will solidify so that a solid matrix is formed (Aripin, 2017).

Chitosan and glycerol plasticizer are added to make this biodegradable plastic. The use of chitosan as an additive in making biodegradable plastic will reduce the speed of water absorption, increase the mechanical properties, and reduce the moisture properties of the film. Glycerol as a plasticizer functions to increase elasticity by reducing the degree of hydrogen bonding and increasing the distance between polymer molecules.

Bioplastic that is produced must fulfill these criteria based on the Indonesian National Standard which must meet the criteria of tensile strength, elongation, swelling, thickness, and biodegradation.

Table 1. Bioplastic standart (SNI No. 7188.7:2016)

Thickness (mm)	Swelling (%)	Tensile strength (MPa)	Elongation (%)	Biodegradation (%)
<0,25	<14	>0,39	10-50	100% (60 days)

This research is a study using durian rind cellulose with variable content of glycerol and chitosan. This research was conducted to determine the best conditions for chitosan in a cellulose solution and the addition of glycerol to the mechanical properties of bioplastics produced.

Material and Methods

Material

The main ingredient used in this research is cellulose obtained from durian rind from Cileungsi with a cellulose content of 55.68%. The auxiliary ingredients used are crab chitosan, glycerol, sodium hydroxide, 5% sodium hypochlorite, glacial acetic acid, and distilled water. The tools used in this research were an oven, hot plate magnetic stirrer, stir bar, 60 mesh sieve, Erlenmeyer, measuring cup, analytical balance, glass stirrer, thermometer, volume pipette, measuring flask, watch glass, mold, and beaker glass.

Preparation of durian rind cellulose

Durian Rind Preparation

The durian rind is cleaned and the white inner rind of the durian is cut into small pieces. Then dried using an oven for 2 hours at 100oC. Once dry, the durian rind is blended until smooth and sieved until it passes 60 mesh.

Durian rind delignification

Take 30 grams of dried durian rind. Then it was added using a delignification solution, namely NaOH with a concentration of 3%, 300 ml, and heated to a temperature of ±75 ˚C for 2 hours. Next, the solution is heated, then filtered and the residue is taken. Then 300 ml of 5% (v/v) NaOCl was added to the results and heated to a temperature of 80˚C for 10 minutes. After that, it was filtered and the cellulose residue was taken, neutralized, and dried in an oven at 55 ˚C for 12 hours.

Making bioplastics from durian peel cellulose

Bioplastics are made using the phase inversion method. Cellulose is dissolved in water and heated at a temperature of 85˚C and a speed of 600 rpm until thickened for 1 hour. Add chitosan dissolved in acetic acid solution (w/v, 1% acetic acid solvent) and heat at 85˚C for 15 minutes. The chitosan and cellulose mixture was then added with glycerol and stirred at 50˚C for 15 minutes. After that, the solution is printed in a mold. Then dried using an oven at a temperature of 55˚C. After drying, release the bioplastic from the plate. Characterization of bioplastic sheets was carried out using tensile tests, elongation tests, swelling, and biodegradation tests.

Tensile strength test

Tensile strength testing is carried out using a Universal Testing Machine (UTM) tensile test tool so that the tensile strength of the material can be determined to withstand the load or mechanical force applied until it breaks/damages. The tensile strength test (σ) is a comparison between the maximum stress (Fmax) and the surface area (A).

$$\sigma = \frac{F\ max}{A}$$

Elongation test

The results of tensile strength tests carried out using a Universal Testing Machine (UTM) are the basis for testing the elongation of bioplastics. According to (Shura, 2020), bioplastic elongation (ε) can be found by comparing the increase in length (Δl) with the original length (lo).

$$\varepsilon = \frac{\Delta l}{l_o}\ x\ 100\%$$

Swelling test (Water absorption)

The method used for the water resistance test (swelling) is based on the method used by Lazuardi and Cahyaningrum (2013). Bioplastic samples were cut into 5x5 cm sizes and weighed initially (W0). After that, the sample was put into a beaker filled with distilled water. Samples were left for 1 minute. After 1 minute, take the sample and remove the water on the surface of the sample using a tissue, then the sample is weighed again to obtain the final weight (W).

$$\text{Swelling (\%)} = \frac{W-W_o}{W_o}\ x\ 100\%$$

Biodegradation test

Biodegradation testing is carried out by utilizing soil microorganisms as assistants in the degradation process, which is called the soil burial test technique. Samples were weighed as initial weight (M_o), planted in soil, and left exposed to open air then observed every 2 days for 12 days then weighed as final weight (M_1).

$$\%Weight\ loss = \frac{Mo - M1}{Mo} x100\%$$

Results and Discussion

Analysis of cellulose content from delignification of durian rind

The results of durian rind delignification carried out in the research showed that the cellulose content was 55.6792%; hemicellulose 26.94175% and lignin 17.37905%. The cellulose content of durian rind obtained is by the content obtained from the literature, namely in the range of 50-60%, but it is not optimal, as in research conducted by Ana in 2015 which obtained a durian rind cellulose value of 60.45%. Differences in cellulose content can be caused by differences in the content of the durian fruit rind itself or the delignification process, both time and concentration of the cooking solution, which is less than optimal.

Table 2. Cellulose, hemicellulose, and lignin content from delignification of durian rind

No	Component	Research Result (%)
1	Cellulose	55.6792
2	Hemicellulose	26.94175
3	Lignin	17.37905

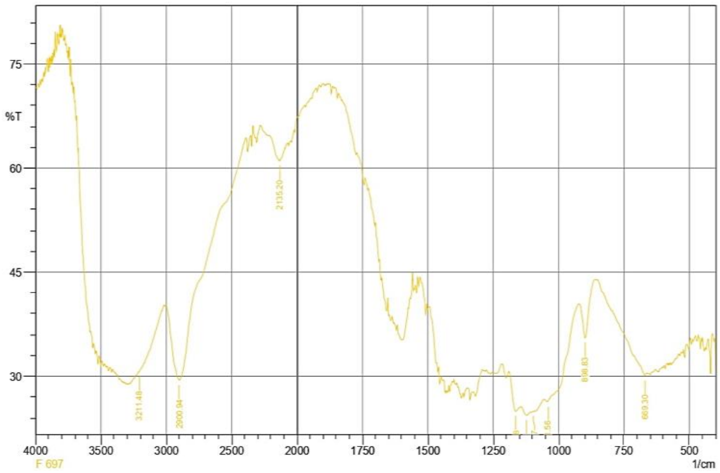

Figure 2. Durian rind cellulose FTIR Test Results

Table 3. Comparison of FTIR Wave Numbers of Durian Rind Cellulose (Abderrahim et al., 2015)

Functional Group	Wavenumber (cm^{-1})	
	*Commercial Cellulose	Research Result Cellulose
O-H Stretching	3391	3211,48
C-H Stretching	2906	2900,94
C-O Stretching	1061	1041,56
B-Glycosidic Bond	898	898,3

From the FTIR spectrum of commercial cellulose, the absorption band at 3391 cm^{-1} was determined to be the hydroxyl group stretching. The bands at 2906 cm^{-1} and 1373 cm^{-1} are assigned to the strain and deformation vibrations of the C-H group in glucose units. The absorption band at 898 cm^{-1} is characteristic of the β-glycosidic bond between glucose units. The band at 1061cm^{-1} was assigned to the -C-O-function group of secondary alcohols and ethers present in the backbone of the cellulose chain (Abderrahim, 2015). The results obtained between commercial cellulose and research cellulose are quite close, but not the same. This happens because of differences in the content of the durian fruit rind itself or because the delignification process is less than optimal.

Durian rind cellulose bioplastic swelling test

Figure 3 shows the effect of chitosan and glycerol concentrations on water absorption capacity. Testing for water resistance on bioplastics is carried out using the water absorption (swelling) method. The ability of plastic to swell when placed in a solution is known as swelling. Bioplastic preparations are tested for water resistance. The higher the water absorption capacity of a bioplastic, the lower the water resistance it produces, and vice versa. This is in line with research conducted by Illing and Satriawan (2019) which found that plastics that have lower water absorption have higher water resistance. Glycerol plasticizer tends to be hydrophilic which will form hydrogen bonds with water molecules because its attraction to water increases (Mashuni, 2021). The more weight of chitosan added, the smaller the water absorption value will be, so the water resistance value will be greater. The hydrophobic nature of chitosan which is not soluble in water causes a higher chitosan weight to reduce its water absorption capacity so that its water resistance value increases (Hayati, 2020).

Table 4. Observation of the effect of chitosan and glycerol concentration on swelling

Chitosan (%)	Glycerol (%)	Initial Weight (gr)	Final Weight (gr)	Thickness (mm)	%Swelling
8%	**1%**	0.6706	0.9122	0.15	36.027
	2%	0.7476	1.1077	0.17	48.167
	3%	0.8483	1.2746	0.18	50.253
	4%	0.7458	1,1658	0.19	56.315
	5%	0.9516	1,5034	0.20	57.987
10%	**1%**	0.9310	1,2487	0.17	34.125
	2%	0.9379	1,3131	0.18	40.004
	3%	0.9407	1,3393	0.18	42.373
	4%	1.0135	1.4837	0.19	46.394
	5%	1.0878	1.6713	0.20	53.640
12%	**1%**	1.1099	1.4325	0.18	29.066
	2%	1.1074	1.5344	0.19	38.559
	3%	1.0900	1.5175	0.20	39.220
	4%	1.1258	1.6102	0.21	43.027
	5%	1.2047	1.7392	0.22	44.368
14%	**1%**	1.1288	1.4417	0.19	27.720
	2%	1.1933	1.6411	0.20	37.526
	3%	1.2284	1.7043	0.21	38.741
	4%	1.1841	1.6535	0.22	39.642
	5%	1.2042	1.7159	0.23	42.493
16%	**1%**	1.1381	1.4239	0.20	25.112
	2%	1.1297	1.5068	0.20	33.381
	3%	1.5006	2.0509	0.21	36.672
	4%	1.5533	2.1395	0.22	37.739
	5%	1.5449	2.1782	0.24	40.993

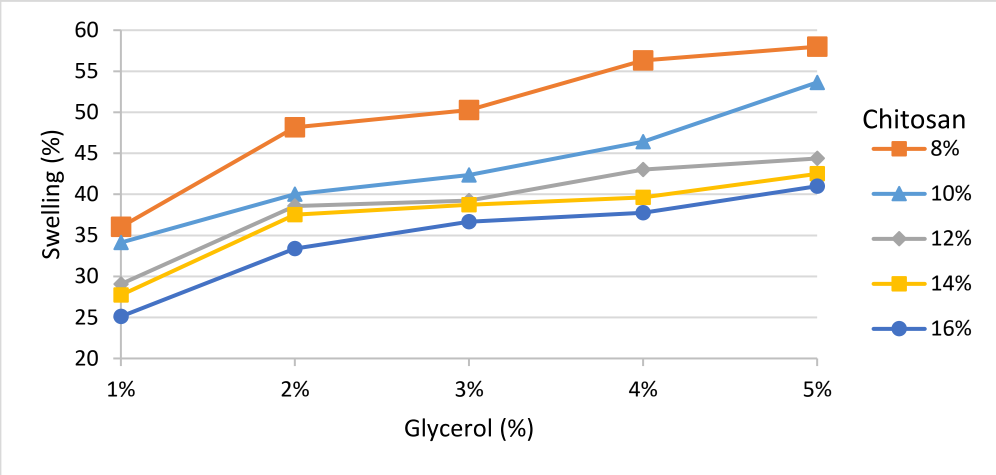

Figure 3. Effect of Glycerol Concentration on Swelling (Water Absorption) at Various Chitosan Concentrations

The composition and volume of the bioplastic solution affect the film thickness. The thicker the film, the more chitosan and glycerol it contains. The difference in concentration of the materials used causes the bioplastic to become thicker, even though the amount of solution poured onto the plate is the same. If there are fewer variations in chitosan and glycerol, the evaporation of the water composition will be greater, which means the bioplastic is thinner. On the other hand, if there are more variations of chitosan and glycerol, the evaporation of the water composition will be less, which means the bioplastic is thicker. This causes the total solids in the bioplastic mixture to increase, resulting in a thicker bioplastic solution. This increases the total solids of the bioplastic mixture, resulting in a thicker bioplastic solution (Mashuni, 2012). The thicker the bioplastic solution poured onto the plate, the thicker the resulting film. This is because more ingredients are added.

The water absorption value obtained in this research still does not meet SNI standards, namely <14%. The smallest water absorption value was obtained by the bioplastic variation of 16% chitosan and 1% glycerol, namely 25.112% with a thickness of 0.20 mm, and the largest water absorption value was obtained by the bioplastic variation of 8% chitosan and 5% glycerol. The thickness of the bioplastic obtained in this study meets SNI standards with a thickness of <0.25 mm with the largest bioplastic thickness obtained from the bioplastic variation of 16% chitosan and 5% glycerol.

Durian rind cellulose bioplastic tensile strength test

Figure 4 shows the effect of chitosan and glycerol concentration on tensile strength. The highest tensile strength value was obtained by the bioplastic variation of 16% chitosan and 1% glycerol of 1.1603 Mpa and the lowest tensile strength value was obtained by the bioplastic variation of 8% chitosan and 5% glycerol of 0.9761 Mpa. The more chitosan added, the stronger the tensile strength, but if too little glycerol is added, the resulting bioplastic will break easily or be less elastic. According to Sinaga et al. (2014), glycerol can increase the flexibility of bioplastics and reduce the intermolecular strength of bioplastics between polymer chains.

The tensile strength value of the bioplastic obtained is by SNI, namely >0.39 MPa. There were fluctuations in the value of the bioplastic variations of 14% chitosan and 4% glycerol with a value of 1.0231 MPa with 14% and 5% chitosan with a value of 1.0368 MPa. This can occur due to the imperfect distribution of each component in the bioplastic film.

Table 5. Observation of the effect of chitosan and glycerol concentration on tensile strength

Chitosan (%)	Glycerol (%)	Mass (kg)	Tensile Strength (Mpa)
	1	52,4	1,0270
	2	51,3	1,0055
8%	**3**	50,2	0,9839
	4	50	0,9800
	5	49,8	0,9761
	1	54.6	1.0702
	2	52.2	1.0231
10%	**3**	51.7	1.0133
	4	50.8	0.9957
	5	50.0	0.9800
	1	56.8	1.1133
	2	55.0	1.0780
12%	**3**	53.2	1.0427
	4	51.7	1.0133
	5	50.2	0.9839
	1	58.0	1.1368
	2	56.6	1.1094
14%	**3**	54.4	1.0662
	4	52.2	1.0231
	5	52.9	1.0368
	1	59.2	1.1603
	2	58.2	1.1407
16%	**3**	57.2	1.1211
	4	56.4	1.1054
	5	55.6	1.0898

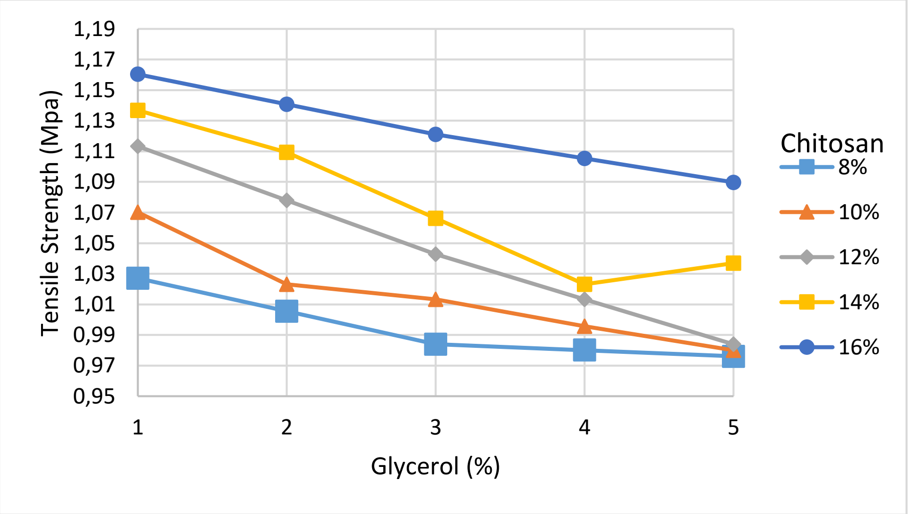

Figure 4. Effect of Glycerol Concentration on Tensile Strength at Various Chitosan Concentrations

Durian rind cellulose bioplastic elongation test

Table 6. Observation of the effect of chitosan and glycerol concentration on elongation

Chitosan (%)	Glycerol (%)	Length (mm)	Elongation (%)
8%	**1**	14.25	14.25
	2	14.41	14.41
	3	14.57	14.57
	4	15.11	15.11
	5	15.11	15.11
10%	**1**	13.72	13.72
	2	14.16	14.16
	3	14.39	14.39
	4	14.61	14.61
	5	15.08	15.08
12%	**1**	13.19	13.19
	2	13.73	13.73
	3	14.27	14.27
	4	14.54	14.54
	5	14.81	14.81
14%	**1**	12.50	12.50
	2	13.08	13.08
	3	13.42	13.42
	4	13.75	13.75
	5	14.27	14.27
16%	**1**	12.20	12.20
	2	12.72	12.72
	3	13.23	13.23
	4	13.58	13.58
	5	13.92	13.92

Figure 5 shows the effect of chitosan and glycerol concentrations on elongation. The elongation value of bioplastics shows the opposite of the tensile strength value of bioplastics. As the chitosan concentration increases, the elongation value decreases. With glycerol, bioplastics can become more flexible and reduce the intermolecular strength between polymer chains. The tensile strength increases with chitosan concentration, but the elongation value decreases. The increasing concentration of chitosan has an impact on increasing tensile strength, conversely, the elongation value decreases (Aripin et al., 2017).

The tensile strength value of the bioplastic obtained is by SNI, namely 10-20%, where in this study the highest elongation value was obtained for the 8% chitosan and 5% glycerol variation bioplastic, 15.11%, and the lowest elongation value was obtained for the chitosan variant bioplastic, 16%. and 1% glycerol of 12.20%.

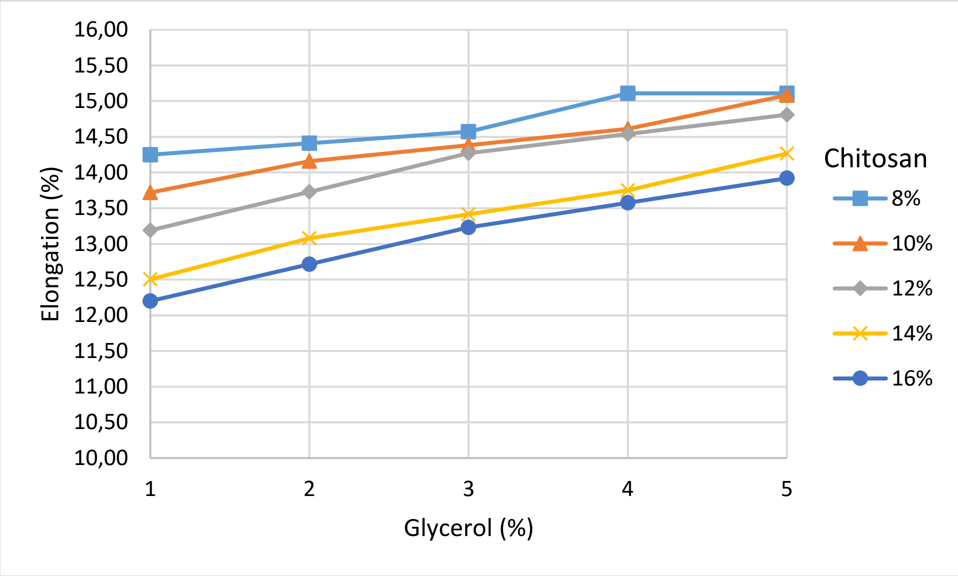

Figure 5. Effect of glycerol concentration on elongation at various chitosan concentrations

Durian rind cellulose bioplastic biodegradation

Table 7. Observation of the Effect of Chitosan and Glycerol Concentration on Biodegradation

Chitosan (%)	Glycerol (%)	Initial Weight (gr)	Final Weight (gr)	%Biodegradation	Biodegradation Time 100% (Day)
8%	**1**	0.6142	0	100.00	12
	2	0.8356	0.2213	73.52	14
	3	0.8692	0.3046	64.96	14
	4	0.7423	0.2685	63.83	16
	5	0.7361	0.3093	57.98	20
10%	**1**	0.8450	0.0021	99.75	14
	2	0.9401	0.3127	66.74	16
	3	0.8303	0.2921	64.82	16
	4	1.0255	0.3981	61.18	18
	5	0.9141	0.4017	56.06	22
12%	**1**	0.9224	0.0057	99.38	14
	2	0.9747	0.3493	64.16	18
	3	1.0182	0.3787	62.81	18
	4	1.0255	0.4102	60.00	20
	5	0.9079	0.4284	52.81	24
14%	**1**	0.9515	0.0096	98.99	14
	2	0.9710	0.3806	60.80	18
	3	1.0182	0.4121	59.53	20
	4	1.0707	0.4914	54.10	22
	5	1.0473	0.5341	49.00	26

To be continued...

	1	1.1058	0.0112	98.99	14
	2	1.1247	0.4483	60.14	20
16%	**3**	1.3047	0.7051	45.96	24
	4	1.3671	0.7903	42.19	28
	5	1.2452	07809	37.29	32

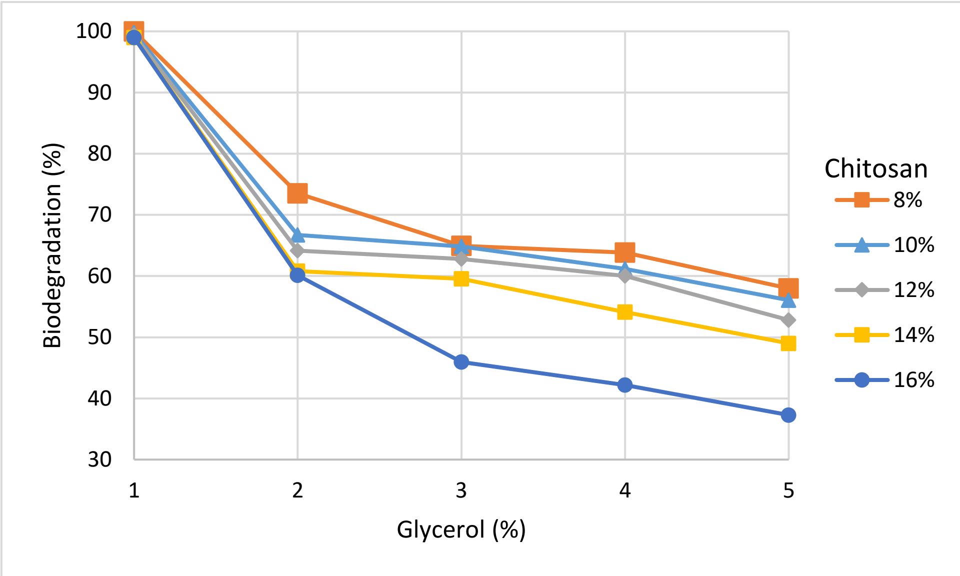

Figure 6. Effect of Glycerol Concentration on Biodegradation at Various Chitosan Concentrations

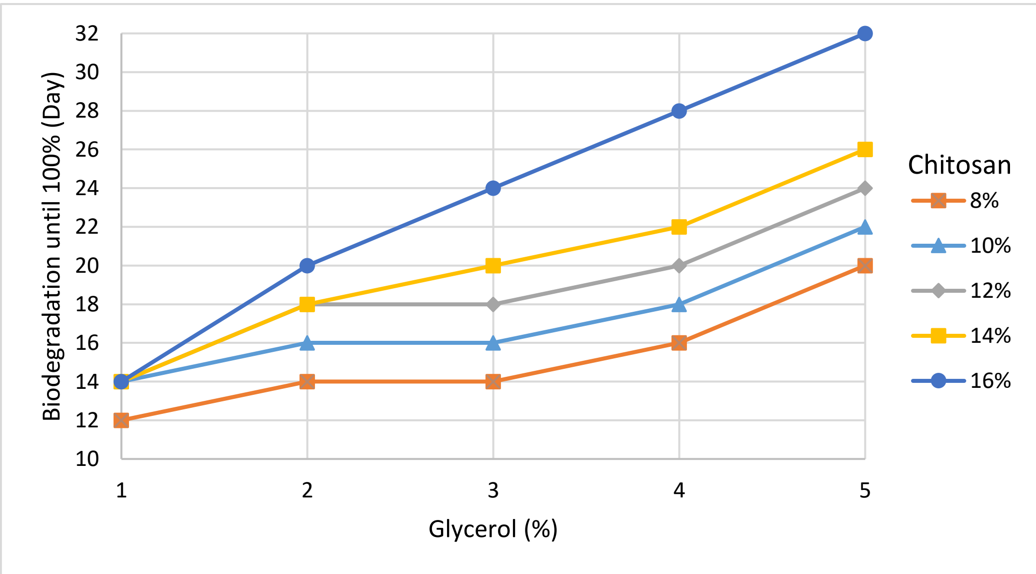

Figure 7. Effect of Glycerol Concentration on Biodegradation until 100% at Various Chitosan Concentrations

Based on Figure 6, it can be seen that on average the bioplastic samples experienced a decrease in their biodegradation ability. It can be seen that the addition of chitosan to the degradation percentage value is inversely proportional. This shows that bioplastic samples decompose more quickly in nature with lower chitosan and glycerol compositions. This is by research by Solekah et al. (2021) which states that the lower the composition of chitosan and glycerol, the faster the bioplastic sample will decompose in nature. The more chitosan, the less biodegradability properties. According to Alam et al. (2018), the hydrophobic and antimicrobacterial properties of chitosan cause damage and mass shrinkage over a longer period.

The highest biodegradation value in this study was obtained by bioplastic variations of 8% chitosan and 1% glycerol with a biodegradation value of 100% in 12 days. Meanwhile, the lowest biodegradation value was found in bioplastics at 16% chitosan and 5% glycerol with a biodegradation value of 37.29% in 12 days and 100% in 32 days. The biodegradation value in this study is appropriate because according to SNI standards, bioplastics must be 100% degraded in 60 days while the lowest biodegradation value is 37.29% in 12 days and 100% decomposed in 32 days.

Conclusion

Based on the results of the research data obtained and the discussions that have been carried out, the following conclusion can be drawn that the best bioplastic results from durian rind cellulose are in the variation of 16% chitosan and 1% glycerol, obtained bioplastic with a tensile strength value of 1.1603 Mpa, biodegradability 98 .99% in 12 days and water absorption capacity of 25.11% and elongation of 12.20%. Bioplastic test results for tensile strength, elongation, and biodegradation are by SNI, while for water absorption capacity they are not by SNI. This shows that the bioplastics that have been obtained need further research regarding the optimum formulation for making bioplastics so that it meet all SNI standards.

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