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SYNTHESIS AND CHARACTERIZATION OF CHITOSAN MEMBRANES OF CASSAVA PEEL *(Manihot esculenta)* AND LERI STARCH COMBINATION AS A BIODEGRADABLE PLASTIC

Sintesis dan Karakterisasi Membran Kitosan Kombinasi Pati Kulit Singkong *(Manihot esculenta)* dan Leri sebagai Plastik Biodegradable

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ABSTRACT
Problems related to landfills of plastic packaging waste are a source of environmental pollution. This research provides innovation in utilizing biodegradable plastic from cassava and leri peel starch with the addition of chitosan. This study aimed to analyze the results of the synthesis and characterization of chitosan membranes with a combination of cassava and leri peel starch and determine the biodegradation time of biodegradable plastic in EM-4 bioactivation. This study characterized biodegradable plastic membranes using test parameters, namely the SEM, FTIR, Swelling, and tensile strength tests. The biodegradation test was carried out by immersing biodegradable plastic membranes in an EM-4 bioactator for 7 days. Based on the results obtained from the characterization of biodegradable plastic membranes, namely the swelling test with concentrations of 5:3, 7:3, and 9:3, with yield values of 62.9921%, 69.8902%, and 71.4286%. The SEM test obtained the best results at a concentration of 7:3. FTIR test on biodegradable plastic membranes containing hydroxyl (O-H) and carbonyl (C-O) groups. The tensile test obtained optimum results at the concentration of biodegradable plastic membranes, namely 9:3. As for the biodegradation test that has been carried out, the start time for degradation is 3 days.

Keywords: *Biodegradable, Characterization, starch, chitosan, cassava peel*

ABSTRAK
Permasalahan terkait dengan timbunan sampah dari kemasan plastik menjadi sumber terjadinya pencemaran lingkungan. Dalam penelitian ini memberikan inovasi dalam memanfaatkan plastik biodegradable dari pati kulit singkong dan leri dengan penambahan kitosan. Tujuan dari penelitian ini adalah untuk menganalisis hasil sintesis dan karakterisasi membran kitosan dengan kombinasi pati kulit singkong dan leri, serta untuk mengetahui waktu biodegradasi yang diperlukan plastik biodegradble dalam bioaktivator EM-4. Dalam penelitian ini melakukan karakterisasi pada membran plastik biodegradable menggunakan parameter uji yaitu uji SEM, FTIR, Swelling, dan tarik regang (tensile strength). Sedangkan uji biodegradasi dilakukan dengan cara perendaman terhadap membran plastik biodegradable pada bioaktivator EM-4 selama 7

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hari. Berdasarkan hasil yang telah didapatkan dari karakterisasi membran plastik biodegradable yaitu pada uji swelling dengan konsentrasi 5:3, 7:3, dan 9:3, dengan nilai hasil yaitu 62.9921%, 69.8902%, 71.4286%. Pada uji SEM didapatkan hasil terbaik pada kosentrasi 7:3. Uji FTIR pada membran plastik biodegradable terdapat gugus hidroksil (O-H) dan karbonil (C-O). Uji tarik regang didapatkan hasil optimum pada konsentrasi membran plastik biodegradable yaitu 9:3. Sedangkan untuk uji biodegradasi yang telah dilakukan mendapatkan waktu mulainya terdegradasi yaitu selama 3 hari.

Kata Kunci: Biodegradable, karakterisasi, kitosan, leri, kulit singkong

INTRODUCTION

Plastic waste often becomes a world problem. The problems caused lead to environmental pollution and even cause disease in living creatures around it (Rahmayani & Aminah, 2021). Microorganisms cannot degrade plastic waste in the ground. These cases to made up of plastic waste to accumulates and last a long time if left without recycling (Renilaili, 2019). To overcome this problem, the one of technologies that can be utilized is to develop biodegradable plastic. Biodegradable plastic is a biologically degradable biopolymer produced by microorganisms (Sasria, 2020).

Several research results reported using natural materials as a basis for making biodegradable plastic. One of them is research conducted by Ezenkwa et al (2022) the research was conducted with tapioca powder and the addition of chitosan. The results showed a composite film with a chitosan content of 8 ppm had a higher degradation rate, within 24 weeks. However, in this study, the time required for the tapioca membrane with the addition of chitosan to degrade was still quite long. This research provides an alternative biodegradable plastic technology that utilizes starch from cassava peel combined with leri *(rice-washing wastewater)*.

In this study, we used leri because leri has advantages that were still limited to the general public, so many people threw it away without knowing that it still contained starch. Leri contains around 76% starch-type carbohydrates in the rice (Layudha et al, 2017). Meanwhile, cassava peel still contains quite a lot of starch. Cassava peel starch has a chemical component of around 44-59%. Starch is a mixture of two main glucose polymer components, linear chain amylose molecules, and branched glucose polymer amylopectin (Muzaffar et al., 2020). These can be utilized in making biodegradable plastic films. Apart from that, cassava peel contains calcium oxalate, fiber, tannin compounds, and glucose (Suryati et al, 2016).

The addition of cassava peel and leri starch is useful as a basic ingredient for making biodegradable plastic because that material speeds up the biodegradation process. On the other hand, a high and optimal water absorption capacity can be an ideal medium for the growth of decomposing microorganisms (Syuhada et al, 2020). In this research, variations in the concentration of chitosan and starch from cassava peel and leri will be carried out. This research aims to obtain the right and effective formulation for developing biodegradable plastic that is environmentally friendly and has the property of decomposing quickly in a relatively short time. As well as being an alternative used as a medium for packaging food and drinks as well as plastic bags which are easily decomposed by microorganisms in nature.

MATERIAL AND METHODS

Place And Time of the Research

This research was conducted at the Science Laboratory, Faculty of Health and Science, Universitas Dhyana Pura, Bali. The research agenda included the production of cassava and leri skin starch, preparation of cassava and leri skin starch solutions, creation of chitosan solutions, and fabrication of biodegradable plastics. Additionally, water swelling tests and biodegradation tests of the biodegradable plastics were performed. Characterization of the biodegradable plastic membranes involved FTIR functional group testing at the Universitas Gadjah Mada Testing Laboratory, observation of the membrane pore structure using SEM at the MERO Foundation in Karangasem, Bali, and tensile testing at the Universitas Diponegoro Testing Laboratory. This research was conducted from November 2022 to February 2023.

Tools and Materials

In this research, the ingredients included 500 grams of cassava peel, 1000 ml of leri, 1000 ml of distilled water, 150 ml of glycerol, 100 grams of Chitosan BCCF3856 Sigma-Aldrich, 100 ml of acetic acid (CH_3COOH), and the EM-4 bioactivator. The tools used in this study consisted of a digital oven, an analytical balance, a hot plate, a 100 mesh sieve, a desiccator, an FT-IR spectrophotometer, a Scanning Electron Microscope (SEM), a Stograph VG 10-E tensile strength testing machine, and various other tools. Additional supporting tools included glass and plastic equipment.

Methods

Making Cassava Peel and Leri Starch

To produce starch from cassava peel and leri, 1,000 grams of cassava peel are thoroughly washed under running water. Once cleaned, the cassava peel is soaked in distilled water for 15 minutes. Following this initial soaking, the peel is soaked again for 24 hours and then ground (Prameswari et al, 2022). Next, 2000 ml of leri is blended with the ground cassava peel until the mixture is smooth (Layudha et al, 2017). The blended solution is then filtered, and the filtrate is allowed to settle for 24 hours. The resulting precipitate is dried under direct sunlight for one to two days. Subsequently, the starch is further dried using an oven set at 70°C for 30 minutes. Finally, the oven-dried starch is sifted to remove any remaining impurities (Alfian et al, 2020).

Preparation of Cassava Peel and Leri Starch Solution

Cassava peel and leri starch solutions were prepared at varying concentrations of 5%, 7%, and 9%, as referenced from the research by Fathanah et al. (2017). To create the 5% concentration solution, 5 grams of cassava peel and leri starch were weighed and placed into a 250 mL Erlenmeyer flask, then dissolved in 100 mL of distilled water. The solution was homogenized for 25 minutes on a hotplate at 80°C. The same procedure was applied to prepare solutions with concentrations of 7% and 9%, as well as a 10% concentration as reported by Adil et al. (2020).

Biodegradable Plastic from Chitosan combination with Cassava Peel Starch and Leri Preparation

The stage of making biodegradable plastic was carried out by dissolving chitosan solution and starch solution in a ratio of 6:4. A total of 30 mL of chitosan solution was added to 20 mL of starch solution. Then glycerol with a concentration of 1% was added. The solution was homogenized on a hotplate at 85°C for 25 minutes. The solution was poured into a 20 x 20 cm^2 glass mold. The mold was dried at 60°C for 5 hours in an oven. The formed membrane was removed from the mold and then put into a desiccator for 24 hours (Adil et al, 2020).

Functional Group Analysis

Functional group characterization was performed using FT-IR spectroscopy (Shimadzu, Japan). For this analysis, 1-2 mg of the membrane sample was weighed and mixed with 200 mg of pure KBr powder. The mixture was then homogenized until thoroughly blended. Subsequently, the homogenized mixture was placed in a mold and subjected to pressure using a mechanical pressure device (Dewi, 2019; Permatasari et al, 2022).

Scanning Electron Microscope (SEM) Analysis

The membrane samples were initially cut into small pieces and mounted on carbon tape. The SEM analysis of the biodegradable plastic membrane samples was conducted at the MERO Foundation Karangasem Bali SEM Laboratory (Maladi, 2019).

Swelling Test

The swelling test of the bioplastic membrane will be conducted at the Basic Science Laboratory of Dhyana Pura University. The test involves measuring the weight difference of the membrane before and after immersion in distilled water. Initially, the membrane is cut to a size of 3×3 cm and weighed (W0). It is then placed in a 100 ml beaker containing 50 ml of distilled water and soaked for 10, 20, and 30 minutes. After the soaking period, any remaining water is removed, and the membrane is weighed to determine its wet weight (Wb). The swelling ratio is calculated using the following equation (Alfian et al, 2020).

$$\%WU = \frac{Wb - W0}{W0} x100\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{(1)}$$

Membrane Tensile Test

The membrane was initially cut to a size of 0.1 mm. A strain rate of 10 mm/minute was applied, with a tensile strength of 100 N. This tensile test was conducted at Diponegoro University (Widiasih, 2017).

Biodegradation Test of Biodegradable Plastics

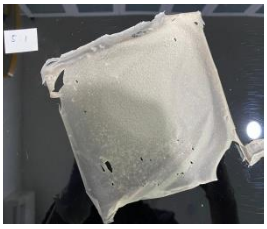
Biodegradation tests were conducted at the Science Laboratory of the Biology Study Program at Dhyana Pura University. The procedure involved cutting the biodegradable plastic into 3x3 cm squares and placing them in a petri dish with EM-4 bioactivator. Observations of the changes in the plastic were made from day 1 to day 7. (Alfian et al, 2020).

Data analysis

The results of the synthesis of chitosan membranes, a combination of cassava peel and leri starch, were then characterized. Characterization results include functional group analysis, SEM analysis, swelling test, and tensile test. This data will be analyzed descriptively and quantitatively.

RESULTS

Based on the results obtained from the nine variations of sample concentrations, the plastic membrane with the best results was at a concentration of 7:3. Judging by the texture and surface appearance, the 7:3 concentration biodegradable plastic membrane is more elastic when pulled by hand. This elasticity can be attributed to the amylose and amylopectin bonds in starch, which are able to bond with the hydrogen bonds in chitosan, forming a plastic membrane with an elastic and strong surface structure. Varying concentrations of cassava and leri peel starch, as well as adding variations in chitosan concentration, were carried out to determine the effectiveness of starch concentration with the addition of chitosan as a bioplastic reinforcement. The addition of 3% chitosan and 1% glycerol provides an optimum concentration for bioplastics to maintain texture when used as packaging. However, the results of this bioplastic synthesis require supporting analysis, such as physical and mechanical characterization, to validate the test results (Dasumiati et al, 2019).


(A)


(B)


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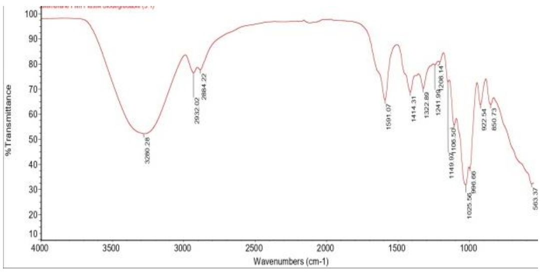
Figure 1. The results of the synthesis of a starch-biodegradable plastic membrane cassava and leri skins with the addition of chitosan in proportions concentration (A) 5:1, (B) 5:2, (C) 5:3, (D) 7:1, (E) 7:2, (F) 7:3, (G) 9:1, (H) 9:2 dan (I) 9:3.

The functional groups contained in this biodegradable plastic membrane show the presence of hydroxyl groups, characterized by O-H bonds. The O-H bonds absorbed in biodegradable plastic membrane samples exhibit different wavelengths depending on the environment. This bond is easy to identify as an acid because it produces a wide peak in the wavelength range of 2500-3300 cm^{-1}. Figure 2 below shows the FT-IR spectrum of biodegradable plastic membranes from cassava and leri peel starch with varying concentrations of chitosan.



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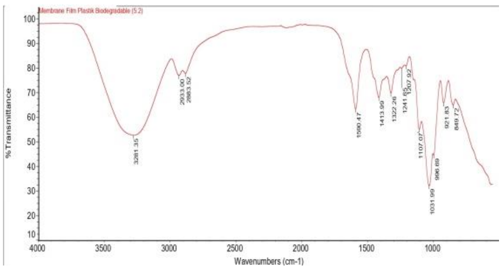

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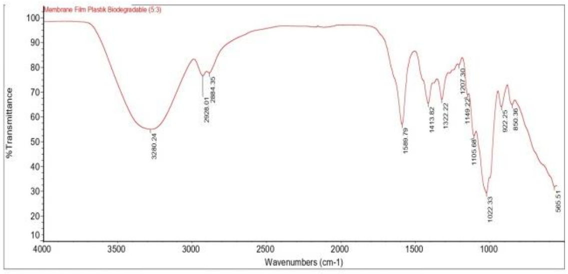

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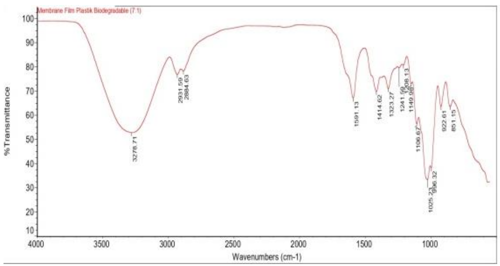

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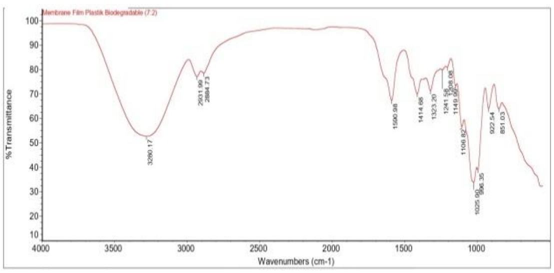

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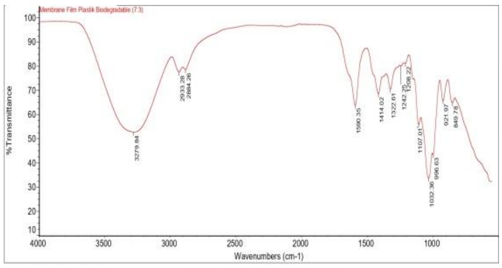

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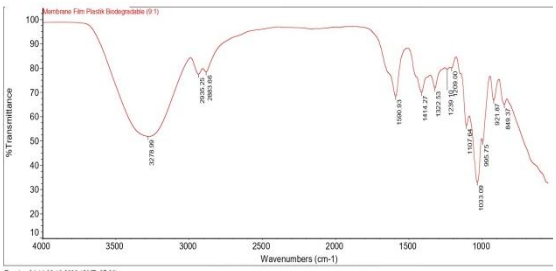

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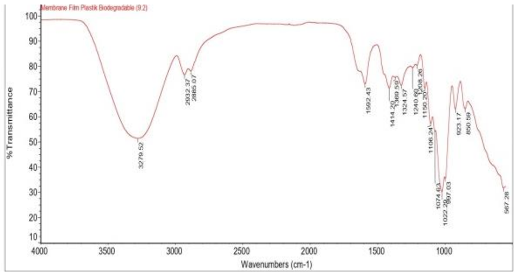

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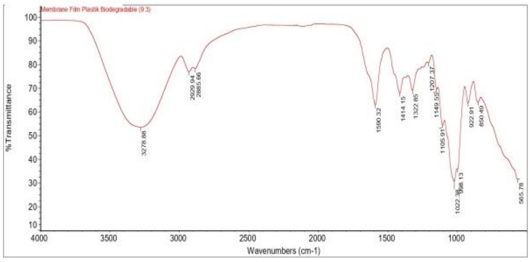


(I)

Figure 2. FT-IR spectrum of biodegradable plastic membranes from cassava and leri peel starch with the addition of chitosan with concentration ratios (A) 5:1, (B) 5:2, (C) 5:3, (D) 7:1, (E) 7:2, (F) 7:3, (G) 9:1, (H) 9:2 dan (I) 9:3.

Based on the FT-IR analysis results, there is also a C-O ester carbonyl functional group, resulting from the breaking of the glycosidic bond chain in the form of a carbonyl group and an O-H hydroxyl group at the end of the amylose or amylopectin contained in cassava and leri peel starch. The C-O ester functional group was identified in the wavelength range of 1300-1000 cm-1 (Nandiyanto et al, 2019). This group with similar wavelength absorption is also caused by the addition of chitosan in the manufacture of biodegradable plastic. The presence of the C-O ester group indicates that biodegradable plastic can break down easily. The C-O ester functional group is hydrophilic, allowing water molecules to cause microorganisms to enter the biodegradable plastic membrane matrix, leading to its degradation in the environment (Maneking et al, 2020).

Table 1. Interpretation of FT-IR spectra of biodegradable plastic membranes from cassava and leri peel starch with the addition of chitosan

Samples	Wavelength (cm^{-1})			
Interpretation	O-H	C-O	C-H alkane bending	C-H alkene stretching
5:1	3280.28	1241.99	1414.31	2932.02
5:2	3281.35	1241.65	1413.99	2933.00
5:3	3280.24	1207.30	1413.82	2928.01
7:1	3278.71	1241.59	1414.62	2931.59
7:2	3280.17	1241.58	1414.68	2931.99
7:3	3279.84	1242.25	1414.02	2933.28
9:1	3278.99	1239.10	1414.27	2935.25
9:2	3279.52	1240.60	1414.20	2932.37
9:3	3278.88	1207.37	1414.15	2929.94

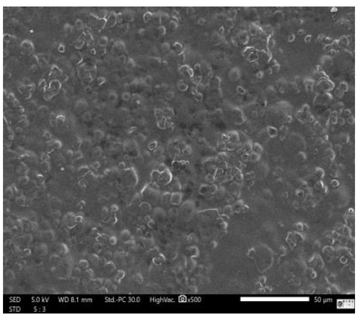
Based on the swelling test results, the 9:3 concentration had the highest water absorption capacity at 71.43%. The lowest percentage of water absorption occurred at a 9:1 concentration, with a value of 23.06%. Meanwhile, the concentrations of 5:3 and

7:3 achieved results of 62.99% and 69.89%, respectively. This occurs due to the higher hydroxyl group content (O-H) in cassava peel starch with the addition of leri, leading to higher water absorption (H_2O). The higher the starch concentration, the greater the water absorption capacity of the biodegradable plastic membrane. Greater water absorption leads to lower resistance and faster disintegration, while lower water absorption results in higher resistance to water and slower disintegration (Lindriati et al, 2021). According to the Indonesian National Standard (SNI) for bioplastics, the swelling test percentage should be 99%. However, the absorption capacity in this study ranges from 23.06% to 71.43%, not meeting the SNI standard 7188.7:2016 (Simarmata et al, 2020).

Table 2. Biodegradable plastic swelling test results

Sample Concentration (Starch: Chitosan)	Dried Membrane Mass (gr)			Wet Membrane Mass (gr)			Swelling Test Result (%)
	M1	M2	M3	M1	M2	M3	
5:1	0.0328	0.0320	0.0351	0.0415	0.0405	0.0488	30.9309
5:2	0.0418	0.0417	0.0405	0.0636	0.0644	0.0615	52.8226
5:3	0.0516	0.0441	0.0440	0.0844	0.0789	0.0644	62.9921
7:1	0.0252	0.0295	0.0280	0.0305	0.0389	0.0378	29.6252
7:2	0.0503	0.0511	0.0574	0.0667	0.0698	0.0755	33.5013
7:3	0.0586	0.0731	0.0596	0.0979	0.1288	0.0983	69.8902
9:1	0.0666	0.0794	0.0630	0.0807	0.0956	0.0809	23.0622
9:2	0.0559	0.0506	0.0615	0.0787	0.0789	0.0870	45.5952
9:3	0.0485	0.0452	0.0421	0.0789	0.0779	0.0760	71.4286

In this research, Scanning Electron Microscopy (SEM) tests were conducted using three magnification scales: 500X, 1000X, and 2000X. The results indicated that the starch granule size in the 7:3 concentration sample was smaller compared to that in the 9:3 concentration sample. This difference can be attributed to the longer dissolution time required for the 9:3 concentration, where the higher amount of starch used prolongs the dissolution process compared to the 7:3 concentration sample. A smaller diameter of starch granules is indicative of a superior morphological structure in biodegradable plastics. Upon examining biodegradable plastic membranes at concentrations of 5:3, 7:3, and 9:3, the most favorable results were observed in the 7:3 concentration. This concentration exhibited no pores on the surface of the biodegradable plastic membrane and had a smaller granule diameter compared to the 5:3 and 9:3 samples. Furthermore, the 7:3 concentration maintained a balanced starch mass, facilitating an easier dissolution process among starch, chitosan, glycerol, and distilled water (Fahnur, 2017).



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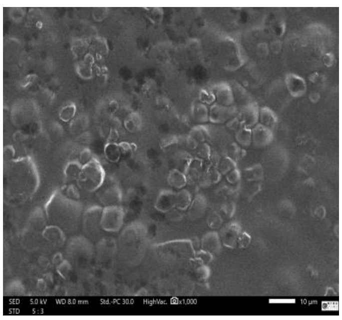

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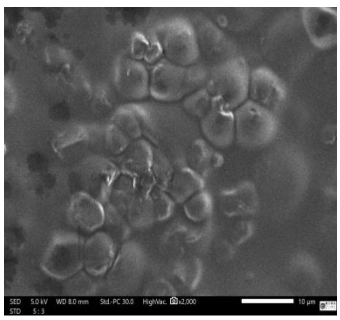

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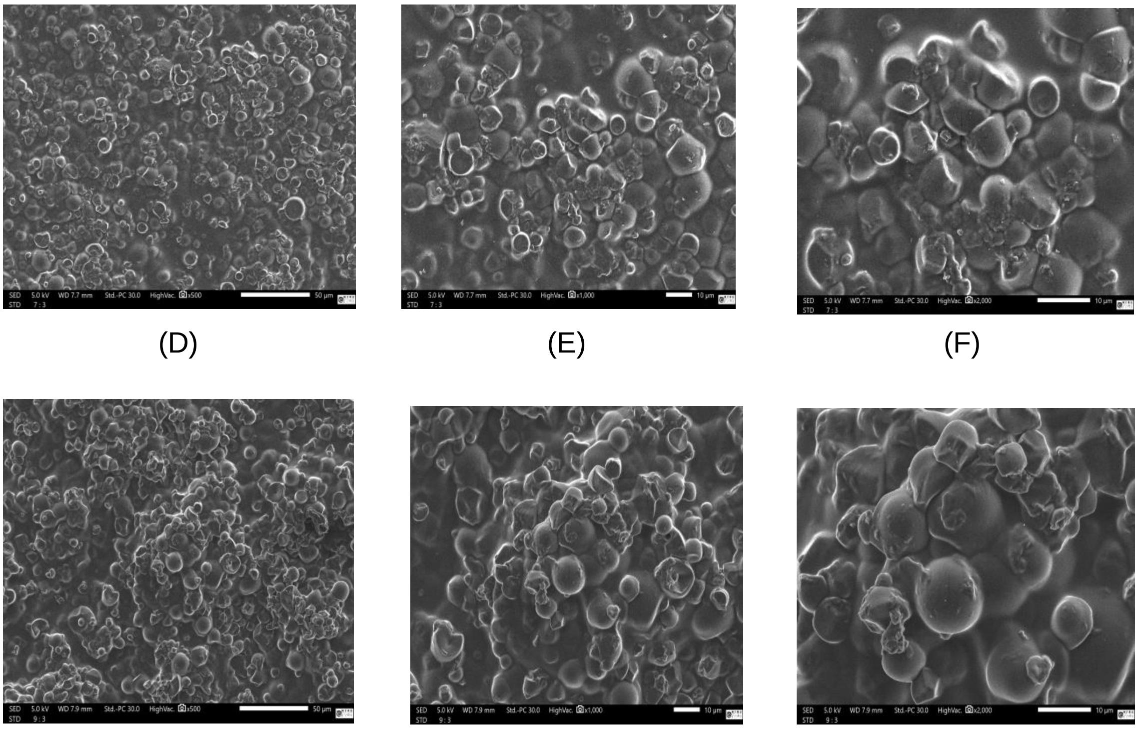


(D) (E) (F)

(G) (H) (I)

Figure 2. SEM characterization results of biodegradable plastic membranes from cassava peel starch and leri with the addition of chitosan in a concentration ratio (A) 5:3 magnification 500X, (B) 5:3 magnification 1000X, (C) 5:3 magnification 2000X, (D) 7:3 magnification 500X, (E) 7:3 magnification 1000X, (F) 7:3 magnification 2000X, (G) 9:3 magnification 500X, (H) 9:3 magnification 1000X, (I) 9:3 2000X magnification identified structural differences biodegradable plastic membrane surface.

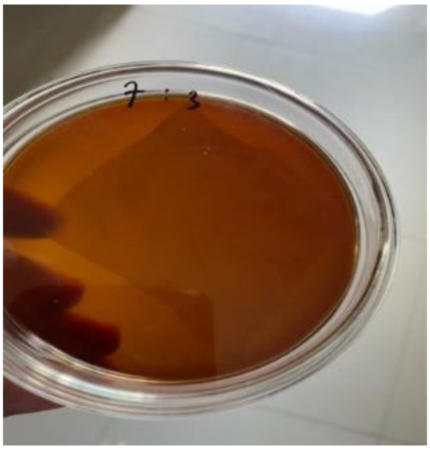
The tensile strength test determines the maximum force or strength required to fracture biodegradable plastic. Biodegradable plastic membranes with high tensile strength values can effectively shield the medium from external mechanical disturbances. The highest tensile strength value recorded in this study was at a concentration ratio of 9:3, incorporating 9% starch and 3% chitosan, yielding a tensile strength of 3.75 MPa. This result can be attributed to the concentration of starch and chitosan, along with the addition of 1% glycerol, which created optimal conditions for tensile strength (Fathanah et al, 2017). The elevated tensile strength is due to the presence of free O-H bonds from chitosan and glycerol, which react and form bonds within the cellulose matrix. Glycerol, being hydrophilic, can easily bind and form amide hydrogen bonds with proteins. However, increasing the concentration of glycerol decreases the tensile strength of the biodegradable plastic membrane (Natalia et al, 2019). Despite these findings, according to the Indonesian National Standard (SNI) 7188.7:2016 for biodegradable plastics, the tensile strength should range from 24.7 to 302 MPa. The tensile strength values observed in this study did not meet the SNI standard (Harsojuwono et al, 2020).

Table 3. Tensile strength test results for biodegradable plastic membranes

Sample	***Tensile Strenght* (Mpa)**	***Elongation* (%)**
5:1	2.02	169.0
5:2	0.48	270.3
5:3	1.58	130.5
7:1	0.42	348.3

Sample	***Tensile Strenght*** **(Mpa)**	***Elongation*** **(%)**
7:2	0.46	316.3
7:3	0.30	125.3
9:1	0.19	51.5
9:2	0.51	119.5
9:3	3.75	111.5

The biodegradation tests are conducted to determine the duration required for biodegradable plastic membranes to decompose in nature. The biodegradation process was assessed by examining the effect of soaking time for biodegradable plastic in an EM-4 bioactivator (Iswendi et al, 2021). EM-4 is known to contain microorganisms such as Lactobacillus sp., Saccharomyces sp., and Actinomycetes sp. (Fahruddin & Sulfahri, 2019). The study evaluated plastic membrane concentrations of 5:3, 7:3, and 9:3, and found that these membranes decomposed completely by the seventh day. Visual observations confirmed the dissolution of the biodegradable plastic membrane in the EM-4 bioactivator. According to the Indonesian National Standard (SNI) Number 7188.7:2016, microbial growth can degrade the surface of biodegradable plastic by more than 60% within 7 days. Consequently, biodegradable plastic made from cassava peel starch with the addition of leri and chitosan meets the SNI requirements for environmentally friendly bioplastics, decomposing in the EM-4 bioactivator within a short period of 7 days (Ismaya et al, 2019).



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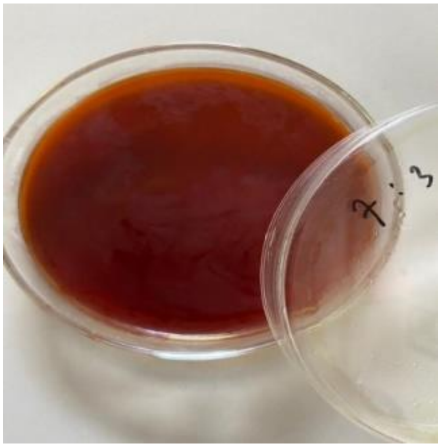

B

Figure 3. Biodegradation results of biodegradable plastic membranes

CONCLUSION

Biodegradable plastic from cassava and leri peel starch with the addition of chitosan and glycerol was successfully synthesized in this research. The membrane characterization results obtained from the swelling test were at concentrations of 5:3, 7:3, and 9:3, with result values of 62.9921%, 69.8902%, and 71.4286%. Meanwhile, in the SEM test, the best results were obtained at a concentration of 7:3. The FTIR test on biodegradable plastic membranes succeeded in identifying the presence of hydroxyl (O-H) and carbonyl (C-O) groups. The tensile tensile test obtained optimum results at the concentration of the biodegradable plastic membrane, namely 9:3. The biodegradable plastic membrane started to degrade on the 3rd day. Then on the 7th day, the biodegradable plastic membrane was completely decomposed in the EM-4 bioactivator. This is characterized by a change in the surface shape of the biodegradable plastic membrane which begins to decompose completely. Further research still needs to be carried out to improve the biodegradation test stage using soil and the addition of an EM-4 bioactivator so that the required degradation time for this bioplastic can be obtained.

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