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Research Article

Synthesis and Characterization of Bioplastic from Macroalgae *Padina australis*

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Abstract

Synthetic plastics are one of the more significant contributors to waste in the environment. Bioplastic is a type of plastic that is environmentally friendly because it is made from biomass. In this study. brown macroalgae. *Padina australis*. is one of the potential raw materials found in large quantities in Indonesian waters and used to produce bioplastics with filtration technique. The aim of this study was to synthesis bioplastics using *P. australis* alginate with glycerol plasticizer. The alginate macroalgae were mixed with glycerol as a plasticizer in the following proportions: 1:15. 1:25. 2:15. and 2:25. with the quality determined through tensile strength measurements. water solubility. and degradation time. On day 12. the tensile strength of *P. australis* bioplastic was 3.24-7.33 MPa. the water solubility was 61.79-65.19%. and the biodegradability rate was 0.1-20.0%. When the macroalgae alginate and glycerol formulation was 2:25. the best bioplastic film produced had a tensile strength of 7.33 MPa and a water solubility of 61.79%. which was lower than the Indonesia National Standard number 7818/2016 for bioplastic products. *P. australis*. however. has the potential to be a promising alternative bioplastic product that contributes to the reduction of petroleum-based plastic pollution in the environment. Furthermore. as a preliminary study. it will be improved to meet industrial standards or find the products that match these characteristics.

Keywords: Alginate, bioplastic, brown macroalgae, *Padina australis*

1. INTRODUCTION

The accumulation of plastic on land and in the oceans is a growing source of concern for society. as it can cause physical problems for organisms and it can also lead to the indirect consumption of microplastic previously ingested by animals and then consumed by humans. Microplastics can cause toxic chemical compounds incorporated into plastics to accumulate in the organism. These chemical substances can accumulate in higher trophic levels. and thus into seafood. creating health issues for humans [1,2].

Synthetic plastic is one of the most significant contributors to environmental waste in Indonesia. According to the Central Bureau of Statistics of the Republic of Indonesia[3]. plastic waste in Indonesia totals 64 million tons per year. with up to 3.2 million tons dumped into the sea. Unfortunately. by 2021. plastic waste will have increased to 66 million tons[4] . This is harmful to the environment and living organisms. Because plastics are difficult to decompose. a solution to create environmentally friendly plastics known as bioplastic is required. Several polymers. including starch. cellulose. chitin/chitosan. protein. and alginate from brown macroalgae. can be used to make bioplastics [5,6] .

New biopolymer-based materials may provide a sustainable solution for replacing synthetic plastic with edible and non-harmful bioplastic. Several studies have already demonstrated that bioplastic can be made from starch. crops. and microalgae [7-9]. However. the use of cultivable marine resources such as macroalgae.

also known as seaweed. is still limited. Widespread use of seaweed is needed because it avoids using land and resources to grow crops; it can also avoid deforestation because seaweed cultivation is done outside or in the laboratory One of them is the use of alginate in the production of biofilms or bioplastics [10.11]. Alginate is a polysaccharide derived from alginic acid. as well as its derivatives and salts [12]. Alginates are anionic linear polysaccharides that make up to 40% of the dry weight of brown seaweeds and have been shown to form edible films.

Research on bioplastics has been done quite a lot. especially with starch raw materials. such as cassava peel starch and chitosan [13]. taro tuber starch [14]. canna starch and chitosan [15]. sago and cassava starch [16]. potato peels and chitosan[17]. as well as other materials such as banana peels [18] and coconut pulp [19]. Only a few studies on bioplastics from native Indonesian macroalgae have been observed. Therefore. the macroalga *Padina australis* was chosen because of its abundant availability in nature. This brown macroalga is one of the many types of macroalgae found widely in Indonesian waters [20]. In addition. *P. australis* has a high carbohydrate content [21]. The carbohydrate content of this material greatly affects its ability to be used as the main material in bioplastics.

The use of macroalgae as raw material for making bioplastics is limited. One of which used alginate from the macroalga *Ulva lactuca* with glycerol as a plasticizer [22] which produced bioplastics with a water solubility of 3.23%. and degradation ability 37-64 % for ten days. Another study used alginate from *Sargassum* sp. mixed with polyethylene [23]. which showed a degradation rate of 16.86% for three days of storage. Research conducted by Akbar [24] tested the tensile strength of red algae *Gracilaria* sp. with a latex mixture that reached the range of 8.14-12.01 MPa. Putri [25] conducted a study using *Euchema* sp. mixed with avocado seeds and cassava starch which showed a degradation rate of 53.54% for 14 days and tensile strength of 1.01 MPa.

The bioplastic manufacture needs a plasticizer to increase the tensile strength of bioplastics and mostly used is glycerol or sorbitol which besides being cheap and easy to obtain. In this study. glycerol was used as a plasticizer. Anward et al. [26] showed that the addition of 20% glycerol and 2% synthetic alginate could optimize the resistance of bioplastics but did not state how much water solubility. tensile strength. and degradation rate. Solak and Dyankova [27] produced bioplastics from alginate and glycerol that can be degraded 30-75% for seven days. Another study showed alginate degradation of *Ulva lactuca* with glycerol to produce bioplastics that degraded 37-64% for ten days [22]. The research by Lim *et al*. [5] resulted in tensile strength of 33.90 MPa and water solubility of 66.27% from tests of *Sargassum* sp. and sago starch with sorbitol as a plasticizer.

Previous studies had shown that the alginate content of *P. australis* was high. reaching 20-49.9% [28]. Besides. the ease and abundance of macroalgae in nature are positive attributes for using it as a raw material to make bioplastics. In this study. 1% and 2% Padina sp. alginate with 15% and 25% glycerol addition was used to produce higher tensile strength and water resistance. The biodegradation test was conducted for 12 days with observations every 3.6.9. and 12 days. The characteristics of bioplastics. such as tensile strength. water-solubility. degradation rate. and functional group tests using Fourier Transform Infrared Spectroscopy (FTIR). were tested in this study. The resulting bioplastic meets Food Grade qualification standards. allowing it to be used in a variety of safe utilities. Because the production of macroalgae-based bioplastics has not been widely developed in Indonesia. this research is critical. Furthermore. bioplastic derived from macroalgae will be one of several environmentally friendly packaging options.

2. MATERIALS AND METHODS

Materials

The materials used were Padina sp. obtained from Thousand Islands. soil. glycerol (Merck). 1%. and 4% calcium chloride ($CaCl_2$) solution (Merck). 5% hydrochloric acid (HCl) solution (Merck). 3% sodium carbonate (Na_2CO_3) solution (Merck). distilled water. and 96% ethanol (Merck).

Alginate Extraction

Alginate of *P. australis* was extracted from 20 g cut samples and soaked in 300 mL of 1% $CaCl_2$ for 18 hours. then rinsed with distilled water. The algae were soaked in 300 mL of 5% HCl solution for 1 hour. then rinsed with distilled water. The algae was Soaked again in 300 mL of 3% Na_2CO_3 for one hour at a temperature of 60-70ºC to form a paste. The solution was filtered then the filtrate was separated. The filtrate obtained was added with 96% ethanol in a ratio of 1:1. The solution was allowed to stand for 24 hours to form a clump of sodium alginate. The sodium alginate obtained was filtered and dried to reduce its water content [29].

Bioplastic Manufacture

Bioplastics are created by dissolving 1% and 2% alginate in 100 mL of distilled water and

stirring until homogeneous. Glycerol was added at 15% and 25% concentrations to each alginate concentration and stirred until homogeneous. Thus. there were 4 variations of ratio of alginate and glycerol: 1:15 (A). 1:25 (B). 2:15 (C). and 2:25 (D). Then. the mixed solution was poured into a 15 x 20 cm glass mold and dried for 4-5 days. For 3 hours. the dried bioplastics were immersed in a 4% $CaCl_2$ solution. The bioplastic was removed from the mold and allowed to dry [26].

Tensile Strength Test

The tensile strength of the bioplastic samples was measured using a tensile measuring instrument. namely the Universal testing machine that refers to ASTM D885. The test was done at a temperature of 23 degrees Celsius with a humidity of 50%. The data obtained directly from the Universal Testing Machine was the change in sample length for any given force. The data obtained were the initial length and the ultimate length. The ultimate length was determined when the sample breaks when pulled. and this data referred to the point of maximum tensile (Fmax). The tensile strength was calculated using equation (1).

$$\text{Tensile strength} = \text{Fmax}/A \times 0.098 \quad (1)$$

where. Fmax: Maximum tensile (Kgf); A: Cross-sectional area (cm^2).

Water solubility Test

The water solubility test was performed by cutting the plastic into 10 x 10 mm and weighing the initial bioplastic sample weight. For 1 minute. bioplastic films were immersed in 10 mL of distilled water. The bioplastic was removed and dried after 1 minute. then the final weight was measured. The immersion was repeated until a constant final weight was obtained [29]. Water solubility is calculated using equation (2).

$$\text{Water solubility (\%)} = (W\text{-}W_0/W_0) \times 100\% \quad (2)$$

where. W_0: initial weight before water immersion (g); W: final weight after water immersion (g).

Biodegradability Test

For the biodegradability test. the soil burial test method developed by Wahyuningtyas and Suryanto [30] was used. The bioplastic films were cut to 10 x 10 mm and buried in soil for up to 12 days. with weight changes measured every 3. 6. 9. and 12 days. The films were collected. cleaned with a tissue. weighed. and returned to the appropriate soil media. The procedure was repeated twice to collect data. The weight loss and biodegradability rate were calculated using equation (3.4).

$$\text{\% weight loss} = (W_1\text{-}W_2)/W_1 \times 100\% \quad (3)$$

$$\text{Biodegradability rate} = (W_1\text{-}W_2)/t \quad (4)$$

where. W_1: initial weight before burial; W_2: final weight after burial. t: time (day)

Moreover. *the Fourier Transform Infrared* (FTIR) spectroscopy was used to confirm the presence of functional groups in samples of macroalgae alginate. Each spectrum was from 4000 to 400 cm^{-1}. This was conducted to ensure that there are only functional groups from macroalgae and nothing else that plays a role in the degradation process.

3. RESULTS AND DISCUSSION

In this study. bioplastics were made from the Indonesian brown macroalga *P. australis* (**Figure 1**) at various ratios of macroalga alginate to glycerol. Tensile strength. water solubility. and the rate of biodegradability of bioplastics were among the physical and mechanical properties studied. Tensile strength was measured to determine how far the material's length increases. Water solubility was measured to determine the percentage of bioplastics' ability to withstand water absorption. The rate of biodegradability was measured in order to estimate how long bioplastics would take to decompose in the environment. **Figure 2** depicts the successful production of bioplastic films from *P. australis* alginate by adding glycerol as a plasticizer in the 4 ratios.

Despite the fact that the smell could not be removed. bioplastic film B had a better physical appearance than the others. The bioplastics all had a fishy odor. which is a problem when using macroalgae for bioplastic. Aside from the appearance of bioplastic. the most important parameters to achieve are its physical and mechanical properties. which are discussed further below.

Figure 1. *P. australis* taken from Indonesian Water Seribu Island

A 1:15	B 1:25	C 2:15	D 2:25
Light brown color, smooth textured surface and slightly stiff sheet, and smells fishy	Lighter brown color, smooth textured surface, smooth and elastic, smells fishy	Slightly dark brown color, the surface is slightly wrinkled, smells fishy	Darker brown color, wrinkled textured surface, smells fishy

Figure 2. Bioplastic film from alginate of *P. australis*

Table 1. Tensile strength of bioplastic

Alginate	Glyserol	Tensile Strength (MPa)	SNI* (MPa)
1%	15%	3.65	13.7
2%	25%	4.60	
	15%	3.24	
	25%	7.33	

*Note: *: Indonesia National Standard for bioplastic bag No. 7818/2014*

Tensile Strength

The highest tensile strength of bioplastic formulation D was 7.33 MPa (**Table 1**). Compared to other studies. the tensile strength of *P. australis* with the addition of glycerol was higher. Langit et al. [31] demonstrated a tensile strength of 0.84 MPa at 5% alginate *Sargassum* and glycerol. Other studies that used raw materials other than alginate had lower tensile strength than this study which tested soybean husks as a basic material for bioplastics and found the highest tensile strength to be 6.5 Mpa [32]. A test with banana peel starch was also conducted. and the bioplastic with the highest tensile strength was 4.26 [33]. Meanwhile. the results of Dasumiati et al. [13] which testing of bioplastics made from cassava starch and chitosan showed better tensile strength results than this study. which was 27.41 and was declared feasible to make plastic bags.

The physicochemical and mechanical properties of alginate gels vary depending on the mannuronic-guluronic (M/G) ratio and the length of the structure. A high guluronic acid content results in stronger gelling properties and a more elastic gel [12]. Although the M/G ratio was not measured in this study. it can be assumed that 2% alginate gels of *P. australis* contained more guluronic acid than 1% concentration.

From **Table 1**. it can be seen that the tensile strength increases with the addition of glycerol. Tensile strength is also improved by a plasticizer. such as glycerol or sorbitol. including the elasticity. flexibility. and extensibility of bioplastics [5]. Glycerol was used as a reinforcement to ensure that the resulting bioplastic was not easily brittle or destroyed. In this study. the tensile strength of the alginate bioplastic *P. australis* with 25% glycerol as plasticizer was greater than 15% glycerol. Yudistriani et al. [32] have produced bioplastics with the use of 20% glycerol and produced a tensile strength value of 6.5 MPa. In this study. the addition of glycerol was only limited to 25%. The addition of glycerol that is too high can pass its saturation point. causing the film to tear easily and will reduce the tensile strength value [33]. The addition of glycerol overcame the brittle nature of the films caused by strong intermolecular forces. Because of the presence of branching bonds in the polymer chain molecules. the addition of glycerol as a plasticizer can increase the mobility of the polymer chain molecules. resulting in increased elasticity and elongation at break [34]. However. if it had passed the saturation point. the film would be easily torn. soft. and the tensile strength value would decrease.

Water Solubility

Table 2 shows that the lower the alginate concentration used. the higher the solubility value. but the higher the alginate concentration. the higher the water resistance. Because alginates are highly

hydrophilic. increasing the number of hydrophilic components leads to a significant increase in the percentage of film solubility [12].

This study discovered that the lowest solubility in 2% alginate was 61.79%. with 38.21% resistance. The higher the alginate content. the easier it is to dissolve. This is supported by the findings of Solak and Dyankova [27]. who discovered 99.13% at a 2.5% alginate concentration without the addition of plasticizers. In comparison to this study. which used 15% glycerol as a plasticizer. this film was 38.21% more water-resistant. The addition of filler to the alginate increases the viscosity of the solution and reduces pore size. resulting in less water accumulation [26].

The presence of glycerol also affects solubility. Glycerol has hydrophilic properties and has enough hydroxyl groups so that it can bind water through hydrogen interactions. The hydrophilic nature of glycerol will increase the solubility of bioplastics in water because the cavity on the surface is getting bigger [31]. This study also found similar results. the higher the glycerol added. the higher the solubility occurred.

Although 25% glycerol shows less percentage of water resistance. the film will still destroy because of the hydrophilic nature of glycerol. besides microbial roles. which is important in degradability when disposed of into the environment. Hydroxyl groups of glycerol have a role in binding with hydrogen interactions [12]. Glycerol is an effective plasticizer because of its ability to soften the film structure. increase the mobility of the biopolymer chain. and improve the mechanical properties of the film by reducing internal hydrogen bonds in intermolecular bonds. As a plasticizer. glycerol improves mechanical properties in bioplastics. such as elasticity. flexibility. and extensibility [34].

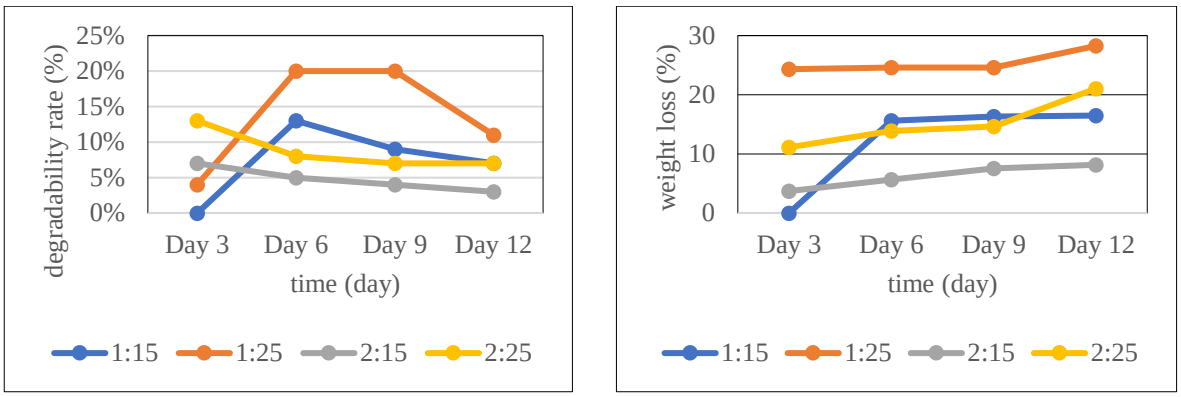
The combination of alginate/glycerol affected the solubility of film from *P. australis*. and this was also proven statistically ($p>0.05$). Moreover. the presence of ions influences alginates' solubility. while their ability to form gels is dependent on the type of cation-cation bond [12]. The addition of calcium in the alginate matrix provides more stability and resistance to the membrane. Unfortunately. this study found lower water resistance compared to Lim *et al*. [5]. Thus. it needs another matrix to increase water resistance and tensile strength.

Based on **Table 2**. film D showed the highest tensile strength and water solubility. The addition of 1% $CaCl_2$ to the gel was not able to enhance the tensile strength of the film. Based on Lim *et al*. [5]. 75% $CaCl_2$ could make high tensile strength film from *Sargassum* alginate to 33.90 MPa. The calcium in the alginate matrix increases the membrane's stability and resistance.

Alginate could also form a rigid and stable gel matrix in the presence of cations. especially Ca^{2+} [5]. Films obtained from sodium alginate with 1%-3% (w/v) calcium chloride solution showed increased tensile strength and elongation properties. and reduced opacity [35]. The addition of starch to the alginate could increase the tensile strength and resistance according to SNI standards [5].

Table 2. Water resistance and solubility of bioplastic

Formulation		Initial weight (g)	Final weight (g)	Water Solubility (%)	Average	Water Resistance (%)	Average
Alginate	**Glycerol**						
1%	15%	0.0042	0.0068	61.90	64.29	38.10	35.71
		0.0042	0.0069	64.29		35.71	
		0.0046	0.0070	66.67		33.33	
		0.0042	0.0069	64.29		35.72	
	25%	0.0044	0.0073	65.91	65.19	34.09	34.81
		0.0044	0.0073	65.91		34.09	
		0.0038	0.0063	65.79		34.21	
		0.0038	0.0062	63.16		36.84	
2%	15%	0.0043	0.0065	51.16	51.79	48.84	48.21
		0.0025	0.0038	52.00		48.00	
		0.0025	0.0038	52.00		48.00	
		0.0025	0.0038	52.00		48.00	
	25%	0.0040	0.0066	65.00	63.45	35.00	36.55
		0.0040	0.0066	65.00		35.00	
		0.0042	0.0068	61.90		38.10	
		0.0042	0.0068	61.90		38.10	



(a) (b)

Figure 3. (a) Biodegradability rate; (b) Weight loss percentage

Biodegradability

Serving as a high-desirability alternative to conventional plastic. tensile strength is an important parameter to achieve. However. the solubility of the film has also been considered as it is related to biodegradability and the spirit to conserve the environment from plastic pollution. The biodegradability rate is to observe the degradability of films when buried.

In this study. the films were buried in the soil for 12 days. and weight loss was measured daily until day 12. Figure 3 depicts the level of biodegradability fluctuating from day to day and tends to decrease on the 12th day. It is due to the decreased work of microorganisms due to reduced nutrition [36]. The average level of biodegradability with 25% glycerol was higher than 15% glycerol. It is related to the hydrophilic nature of glycerol which contributes that the more hydrophilic ions. the faster it degrades.

The weight loss of the film aided the degradation process (**Figure 3**). All of the films lost weight after being buried for 12 days. and the curve showed an increase in movement after 12 days. The best film with the highest weight loss was 28.26% from an alginate/glycerol ratio of 1:25 (B). but this was slower than Solak and Dyankova's studies [27]. which degraded 40-60% sodium alginate films in 12 days. and study of Dewi et al. [22]. which degraded 37-64% in 10 days. As long as the film degrades more easily than petroleum-based plastic. the biodegradability rate. on the other hand. serves as a supplement.

Moreover. according to Adhikari et al. [36]. too large a degradation rate will reduce the durability of the plastic. Langit et al. [31] also found that the higher the alginate concentration. the lower the biodegradation time due to the inhibition of water absorption and the viscosity of the solution. Meanwhile. the homogeneous bioplastics and their dense structure due to physicochemical differences cause the particles contained in bioplastics to be difficult to decompose by microorganisms. Biodegradability is also influenced by the composition and properties of the polymer. the more hydrophilic a polymer will accelerate the degradation process [36]. Based on these studies. it could be stated that with high durability. bioplastic still can degrade afterward with the role of soil microbial.

As mentioned by Solak and Dyankova [27] that alginate films at the end of the period-80th day. were 92.13% loss in weight. This study showed that all films could degrade within 12 days and will continuously degrade. the same as commercial bioplastic (E) (**Figure 4**). All films adsorbed water after being buried in soil and gradually lost their initial shape and structural integrity. It believes that everything decomposed quickly and that a distinct odor of decay occurred [27]. However. beyond the findings of this study. the durability of bioplastic film must be improved.

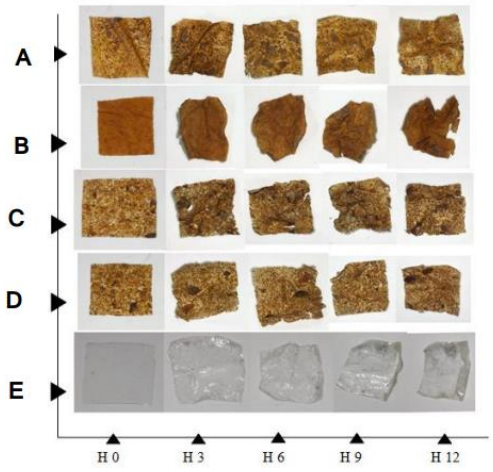


Figure 4. The biodegradation process of bioplastic film for 12 days of burying in soils

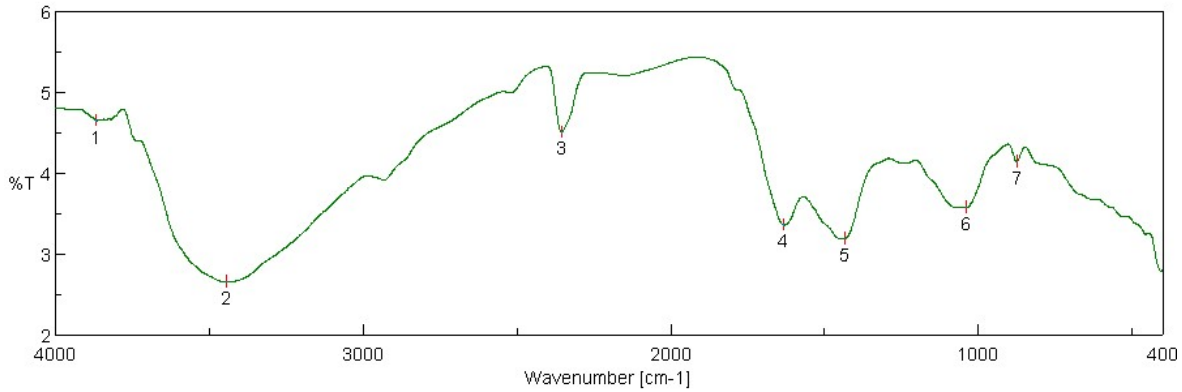


Figure 5. FTIR results for the treatment of 2:25 alginate-glycerol

Moreover. infrared spectroscopy was used to examine the functional groups of sodium alginate extracted from the macroalgae *P. australis*. **Figure 5** depicts the infrared spectrum of sodium alginate extraction. The sodium alginate extraction spectrum revealed absorption peaks at 3441.35 cm^{-1} (2) (O- H stretching vibration). 2354.66 cm^{-1} (3) (C-C stretching vibration). 1632.45 cm^{-1} (4) (C=C stretching vibration). 1436.71 cm^{-1} (5) (C-H stretching vibration). and 1042.34 cm^{-1} (6) (stretching vibration of C-O). The same polymeric bond was discovered at a wavelength not significantly different from other studies [37]. This demonstrates that seaweed plastic has a functional group as a result of a combination of specific functional groups found in its constituent components that play a role in the degradation process.

4. CONCLUSIONS

Padina australis. an Indonesian seaweed. can be used to make the bioplastics film via injection molding with glycerol as the plasticizer. This preliminary study discovered that the higher the seaweed content. the higher the tensile strength of bioplastics made solely from macroalgae. More alginate. on the other hand. increases solution viscosity and decreases pore size. resulting in less water accumulation. In this study. the tensile strength of the film still needs to be improved. specifically by adding environmentally friendly fillers derived from organic wastes such as chitosan waste. agricultural waste. and fruit plant seeds. including native starch. which has been extensively researched but few combine with macroalgae. According to the findings of this study. the alginate-glycerol ratio of 2:25 provided the best value in terms of tensile strength though it was still lower than the SNI standard of 13.7 MPa.

Because the hydrophilic plasticizer used was still ineffective. the film retained its high solubility. As a result of the lower solubility. the mechanical stability of the material should improve. and the polymer should be more cross-linked and resistant to water in the pores of the structure and bonds.

This study's findings were encouraging. but more research is needed to improve the mechanical properties of bioplastics. Composite formulation with other biodegradable polyesters. as well as studies of the effects under various processing conditions. may be a way to achieve that goal and discover suitable new utilities. In the future. this research will be expanded to find a combination that can serve as a high-desirability alternative to conventional plastic for plastic cutlery. Because they completely degrade in soil and are derived from renewable sources. they are environmentally friendly products whose production proves to be an investment in environmental protection.

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