

Synthesis and Characterization of Edible Film Based on Glucomannan from Local Porang Tubers with a Combination of Carrageenan and Sorbitol as Plasticizer

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ABSTRACT

In this study, edible films were made from glucomannan from local porang tubers, carrageenan, and sorbitol as the plasticizer. This research method begins with the making of modified porang flour with three immersion treatments, namely control (A), 10% lime solution (B), and modified lime and mocaf starter fermentation (C) to reduce oxalate levels. Plasticizers were added with variations of carrageenan 2;3;4%, and sorbitol 1;2;3;4; and 5 g. The results showed that the modified lime immersion and fermentation treatment reduced calcium oxalate levels, and the highest yields of glucomannan were 2.58% and 10.07 g. The FTIR analysis results of the isolated glucomannan showed that the spectrum formed by the presence of O-H functional groups, CH₂ groups, C-O-C groups, absorption areas of 805 cm⁻¹ and 872 cm⁻¹ indicated the presence of the main constituent of glucomannan, namely β-pyranose. In comparison, the characteristics of the edible film obtained an average thickness between 0.32–1.50 mm. The highest thickness was obtained in the composition of the addition of 3% carrageenan and 4 grams of sorbitol variation C. The percentage of water resistance of edible film variations A and C ranged from 10.04% - 22.02%. Variations A and C with a composition of 2% carrageenan and 1 gram of sorbitol, the edible film is completely degraded. The surface morphology of the edible film obtained from variation A with a concentration of 2% carrageenan and 5 grams of sorbitol showed a slightly porous surface.

Key words: Calcium oxalates; Edible film; Glucomannan; Plasticizers.

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Introduction

Plastic polymers dominate as raw materials for food packaging. However, their environmentally unfriendly nature increases the risk of health problems due to contamination from various plastic

substances that can migrate into food [1]. Various research has been conducted to reduce the use of plastic, including synthesizing bioplastics from tofu waste [2].

One alternative technology that is more efficient and environmentally friendly is edible film. The edible film is a thin layer formed from polysaccharides, protein, and lipids that can be used as packaging for food products and safely consumed with packaged food [3]. Gelatin is the derivative of protein used in the production of edible film. Gelatin is a protein commonly extracted from animal collagen tissues such as fish, beef, and pork [4]. In Indonesia, the use of gelatin is still a serious consideration due to its halal status, health issues, and high price. Therefore, as a substitute for gelatin in edible film production, konjac glucomannan from porang flour can be used."

The porang tuber (*Amorphophallus oncophyllus*) is quite easily found in Indonesia, including Sumatra. Coarse porang flour contains 15-64% glucomannan, 10-30% starch, 2-5% crude fibre, 5-14% protein, 3-5% reducing sugar, 3.4-5.3% ash, low fat and vitamins. Glucomannan has intermediate properties between cellulose and galactomannan, which can crystallize, form delicate fibre structures, and form elastic gels. This property makes helpful glucomannan and can be further developed into an edible film [5].

The utilization of glucomannan as a raw material for producing the edible film has been carried out in previous research, with variations of glucomannan with glycerol plasticizer and chitosan. The research showed that the edible film has a thickness value of 0.59 mm with a water resistance value of 25.229% and a degradation ability value of 100% but has low water resistance [3].

The addition of plasticizers such as glycerol, chitosan, and sorbitol serve to be able to help improve flexibility, mechanical properties, and water-vapor transmission properties so that it is more resistant to high humidity for the edible film to be produced. In this research, the optimization of edible film production was reported with the addition of carrageenan and sorbitol, which

have better bioplastic quality and characteristics.

Materials and methods

Materials

The equipment used in this study was calibrated glassware, an oven, a 60-mesh sieve, a hot plate stirrer, a centrifuge, a blender, a glass plate, a desiccator, a screw micrometre, an FTIR spectrophotometer, and SEM. The materials used in this study were pro-analysis (p.a.) quality materials such as NH_4Cl , KMnO_4 , H_2SO_4 , HCl , distilled water, 96% ethanol, sorbitol, mocaf starter, carrageenan, filter paper, and porang tubers sampled from the Bayas Jaya Village, Way Khilau District, Pesawaran Regency, Lampung.

Methods

1. Modified Porang Flour Making

Porang tubers that had been obtained were peeled, washed, and sliced to make porang chips. The soaking process was then divided into three treatments, namely the control (making porang flour without any specific addition or modification), modification with lime in a 1:1 ratio soaked for 1 hour, and modification of porang flour with a combination of soaking lime as much as 1:1 for 1 hour, then mocaf starter as a fermentation material for 12 hours. After that, porang chips were dried using an oven at a temperature of 80-100°C. After drying, the chips were ground and sifted using a 60-mesh sieve [6].

2. Analysis of Calcium Oxalate Levels

Potassium permanganate solution 0.1 N was prepared by dissolving 3.16 grams of KMnO_4 powder in distilled water up to a volume of 500 mL. A 0.1 N oxalic acid solution was prepared by dissolving 0.315 grams of oxalic acid dihydrate in 50 mL. 10 mL of the 0.1 N oxalic acid solution was then titrated with the 0.1 N potassium permanganate solution. Before titration, the oxalic acid solution was added with 7 mL of concentrated sulfuric acid and then heated

to a solution temperature of 70°C. The titration was stopped when a pink colour appeared and remained for at least 30 seconds.

3. Pre-analysis Sample Preparation.

One gram of porang flour sample was dissolved in a mixture of 190 mL distilled water and 10 mL 6 M HCl. The mixture is then heated to a temperature of 100°C and diluted with distilled water up to 250 mL before being filtered. The filtrate obtained is ready for analysis.

4. Sample Analysis.

The filtrate obtained is pipetted to 50 mL and then added with 10 mL of 4 N H₂SO₄ solution. The solution is then heated to a temperature of 70°C and titrated with a 0.1 N potassium permanganate solution. The titration was stopped when the solution turned pink.

5. Isolation of Glucomannan

4 g of porang flour was placed in a glass beaker, and then 50 mL of 0.1% NH₄Cl and 100 mL of warm water (75°C) were added. The mixture was stirred using a hot plate stirrer. The resulting precipitate was separated by centrifugation at 2000 rpm. The filtrate was added to 150 mL of 96% ethanol and homogenized using a hot plate stirrer. The resulting slime was filtered using filter paper and dried in an oven. It was then weighed until a constant weight was obtained [6].

6. Synthesis of Edible Film

Carrageenan with various concentrations of 2%, 3%, and 4% (w/v) was dissolved in 100 ml of distilled water. Then, 2 g of isolated glucomannan was added to 100 ml of distilled water and heated on a hot plate stirrer at a temperature of 50°C. Next, 10 ml of the carrageenan solution was slowly added to the mixture. The plasticizer sorbitol was added according to the specified concentration variations of 1 g, 2 g, 3 g, 4 g, and 5 g, respectively, and then the mixture was heated again using a hot plate stirrer at a temperature of 50°C. The solution was then cooled to room temperature to remove air bubbles. The mixture was then dried in an oven at a temperature of 60°C, and after it was dried, it was placed in a desiccator [3]. The edible film was then analyzed for functional groups, resistance, biodegradability, and thickness.

7. Characterization of Edible Film

Edible film that has been produced is characterized using FTIR instrument Shimadzu 820PC to determine the presence of glucomannan functional groups in the edible film, and using SEM instrument model ZEISS EVO MA 10 to know the surface morphology of edible film. Then physical characterization was carried out in the form of water resistance, biodegradation, and thickness to obtain edible films with good quality.

Table 1. Results of analysis of proximate test and modified porang flour yield

No	Parameters	Sample			SNI 7939:2013 (%)
		(A)	(B)	(C)	
1	Ash Content (%)	8,56	15,01	21,23	5-6,5
2	Water content (%)	13,04	14	8,52	≤13
3	Proteins (%)	8,95	4,91	5,57	11-13
4	Fat (%)	1,27	2,86	3,46	-
5	Carbs (%)	57,64	73,70	76,88	-
6	Glucomannan yield (%)	15,12	7,32	10,07	-

Note: (A): Control; (B): Lime Soaking; (C): Lime Soaking + 12 Hours Fermentation

Results and Discussion

This study utilized glucomannan from local porang tubers, which are widely grown by the community. Glucomannan has properties between cellulose and galactomannan, which can crystallize, form fine fiber structures, and form elastic gels to be used as a raw material for making edible films. This research was conducted as a solution for handling plastic waste, which is difficult to degrade by the environment and chemical particles in plastic that can harm health when in direct contact with food. Carrageenan and sorbitol plasticizers were added to obtain better characteristics of edible films than in previous studies.

1. Proximate Test Results and Modified Porang Flour Glucomannan Yield

The first step in isolating glucomannan from porang tubers is to make porang tuber flour. The resulting porang flour is then subjected to proximate analysis and yield calculations. The proximate analysis aimed to determine the properties of the porang flour obtained, from which the glucomannan would be extracted. The results of the proximate analysis and calculation of the yield are presented in Table 1.

Based on Table 1. the results of the research above show that there are differences in the results of the proximate analysis of the 3 treatments given to porang tubers. The analysis results of ash content analysis in control porang flour are closest to the SNI quality standards. A high ash content indicates that many minerals are contained in porang flour, and high minerals in foodstuffs make it difficult for the digestive system to digest. The water content obtained from porang flour from lime soaking and fermentation meets the quality standard of 8.52% from $\leq 13\%$. The water content of food ingredients affects the stability or shelf life of food ingredients. The higher the water content in food, the easier it is for the food to be damaged, both by microbiological damage and by chemical reactions. Analysis of the protein

content showed that the control porang flour had the highest protein content compared to the other two varieties of porang flour. This may be because the porang used has a low protein content. In addition, the modified porang flour also experienced a decrease in protein content due to the non-collagen proteins having been degraded by the lime solution. Amino acids that can bond with a base (lime) are amino acids that have an acid group side chain, such as aspartate acid and glutamic acid. The highest levels of fat and carbohydrates were obtained by porang flour with a modified lime immersion and 12-hour fermentation. From these results, the modification with the best treatment was porang flour with lime soaking and 12 hours of fermentation, where most of the test results met the specified SNI quality limits.

Meanwhile, the yield of glucomannan obtained in the three treatments showed different results. The low yield of modified porang flour was caused by soaking the lime which dissolved the glucomannan content in the porang tubers. The lower glucomannan content in the lime soaking treatment compared to the control porang flour is thought to be due to the alkaline ability of the lime to break the glycosidic bonds in the glucomannan constituents themselves, namely manossa and glucose. Whereas in the lime soaking and fermentation treatment, the yield of glucomannan was higher than the lime soaking treatment, influenced by the mocaf starter fermentation which could withstand the interaction between bases and glucomannan. So that the treatment of porang tubers that has a level of effectiveness in reducing the amount of oxalate and produces a lot of glucomannan is by soaking lime and fermenting for 12 hours.

The isolated glucomannan was then tested using FT-IR instrumentation to identify functional groups. Variation C with lime immersion and fermentation treatment was selected as a sample to be identified in Figure 1.

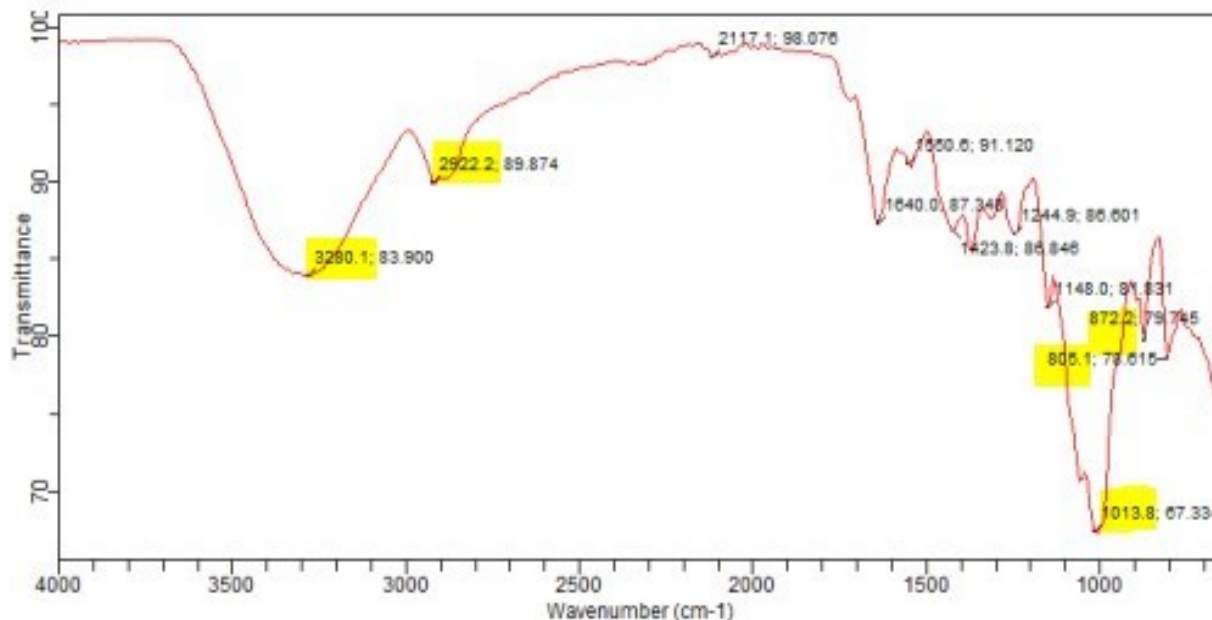


Figure 1. FT-IR spectra of glucomannan powder variation C.

The area measured on the spectrum covers the wave numbers 650–4000 cm^{-1} . In Figure 2, it can be seen in the absorption area of 3280 cm^{-1} . The presence of a peak in the 3000 - 3700 cm^{-1} indicates the presence of the O-H functional group of glucomannan. According to Ruan *et al.* [7], the spectrum of glucomannan is dominated by spectral bands associated with the stretching vibrations of O-H groups and water. The peak in the area of 2922 cm^{-1} shows the vibration of the CH_2 group [8]. Stated that absorption from the carbonyl group ($\text{C}=\text{O}$) was seen in the 1850-1630 cm^{-1} area. Thus, a carbonyl group was identified in the 1640 cm^{-1} region of the tested glucomannan. The presence of the C-O-C group can be seen by the appearance of a peak at the absorption of 1013 cm^{-1} , according to a study conducted by Darmawati *et al.*, [9], where the C-O-C functional group is seen at wave numbers 1019-1016 cm^{-1} . The peaks shown in the absorption areas of 805 cm^{-1} and 872 cm^{-1} indicate the presence of glycosidic bonds from mannose and glucose in the form of β -pyranose as the main constituent of glucomannan compounds. Based on the data generated, it is consistent with previous

research conducted by Nurlela, *et al* [8], who extracted glucomannan from porang flour and obtained glucomannan functional groups, namely (O-H, $\text{C}=\text{O}$, C-O, C-H). It can be concluded that the compound obtained in variation C is glucomannan. It can be concluded that the compound obtained in variation C is glucomannan [10].

2. Calcium Oxalate Content

The determination of the calcium oxalate compound in flour was used by the Permanganometric Titration method [11]. Local porang tubers contain calcium oxalate, which can cause irritation and itching when consumed, so it needs to be minimized. The measurement of oxalate levels in porang tubers is presented in Figure 2.

Based on the graph in Figure 2. That shows that there was a decrease in oxalate levels in porang tubers due to the influence of the treatment given. Lime soaking treatment was able to reduce oxalate levels in porang tubers significantly. Soaking lime (CaCO_3) on porang tubers aims to dissolve calcium oxalate. A decrease in oxalate levels also occurs due to ongoing osmosis

events. Subsequent modifications were made by adding lime and fermenting using a mocaf starter. This fermentation process is considered capable of reducing calcium oxalate levels because the content of lactic acid bacteria in the mocaf starter causes organic acids to lower the pH of the soaking water. So that the presence of calcium oxalate in porang tubers dissolves because the soaking water is acidic [12]. The results

showed a significant decrease in oxalate levels and did not differ much from the lime immersion treatment. However, the lime immersion and fermentation treatments were considered better because they could reduce calcium oxalate in porang tubers and keep glucomannan insoluble in the soaking process so that it still produced high glucomannan yields, as previously described.

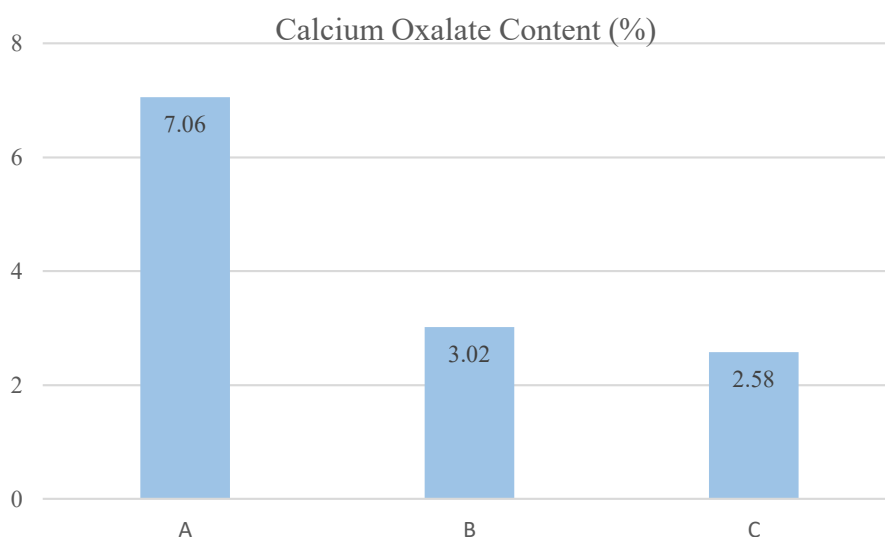


Figure 2. Graph of calcium oxalate levels in control porang flour (A); porang flour modified by lime immersion (B); Porang flour modified by lime immersion and fermentation (C).

3. Characteristics of Edible films

The characteristics of the edible film analyzed in this study were treatment C, namely lime immersion modification and mocaf starter fermentation which produced a higher glucomannan yield than treatment B, namely lime immersion. Meanwhile, treatment A, namely control, was also analyzed as a comparison.

a. Thickness Analysis

Thickness is an important parameter that influences the use of film as a packaging product. The film thickness affects gas permeability. The thicker the food film, the lower the gas permeability and the better the protection of the packaged product. The thickness of the edible film produced was measured with a micrometer screw at five different points to obtain an

average thickness of each edible film in variants A and C. The following results for measuring the thickness of the edible film are presented in Figure 3.

The average value of edible film thickness obtained in this study ranged from 0.32 to 1.50 mm. Based on Figure 3, the highest edible film thickness was obtained with a formulation containing 3% carrageenan and 4 grams of sorbitol, namely variation C. Meanwhile, the lowest edible film thickness was obtained with a formulation containing 2% carrageenan and 1 gram of sorbitol. Increasing the sorbitol and carrageenan concentration greatly affects the edible film's thickness. This is known from the significant increase in viscosity when more sorbitol is added. The increase in total solids, which resulted in the thickening of the edible film, was caused by

an increase in the materials used in making the edible film [3].

b. Water Resistance Analysis

This analysis shows the ability of edible films to absorb water [3]. The effect of adding carrageenan and sorbitol to % water resistance can be seen in Figure 4. Based on Figure 4, the percentage of water resistance for edible film variants A and C varies from 10.04% to 22.02%. The lowest percentage of water resistance was obtained in variant C with 4% carrageenan sorbitol 1 g. Meanwhile, the highest percentage of

water resistance was also achieved in variant C with a composition of 2% carrageenan and 5 g sorbitol. The results indicate that adding carrageenan can increase the solubility of edible films. Carrageenan has hydrophilic properties like glucomannan, so it dissolves easily in water. The higher the sorbitol content, the lower the solubility of the edible film, so the ability of the edible film to bind water increases. Highly soluble edible films can be used in ready-to-eat foods for easy consumption [13].

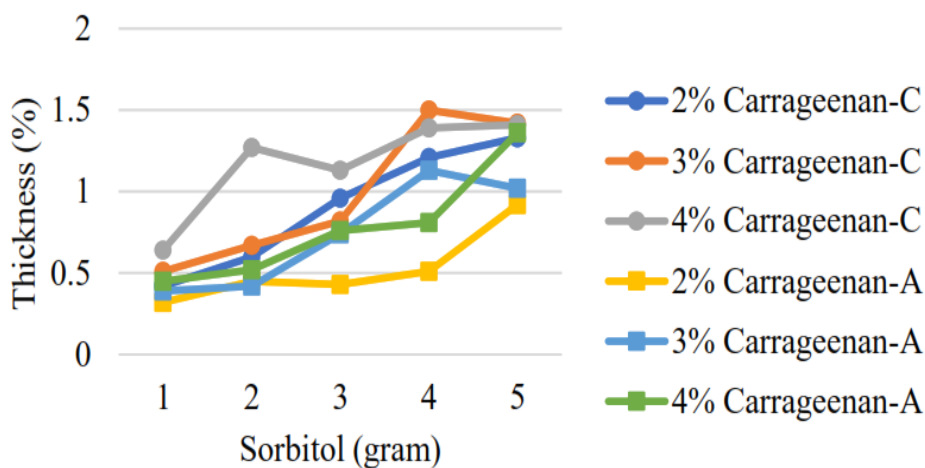


Figure 3. Graph of the relationship between the addition of carrageenan and sorbitol to the thickness of the edible film.

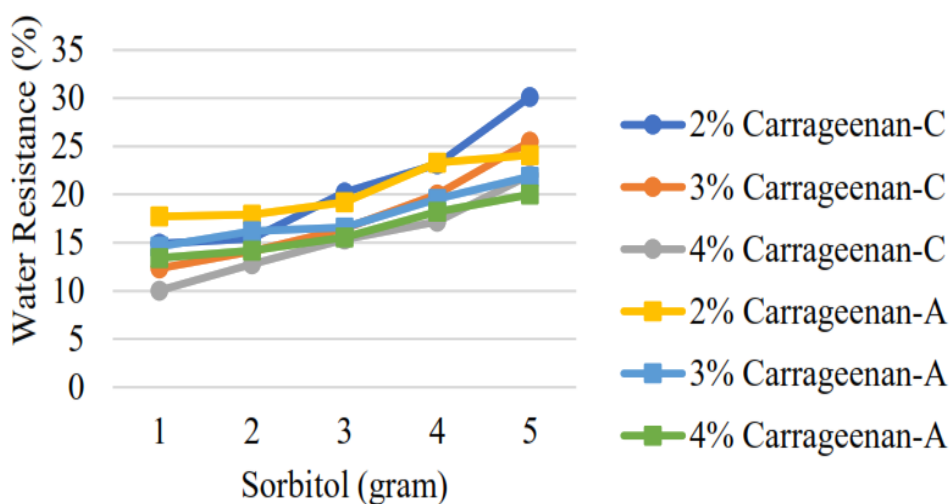


Figure 4. Graph of the relationship between the addition of carrageenan and sorbitol to the water resistance of edible films.

c. Biodegradable Analysis

Biodegradable analysis was carried out to determine the time required for film samples to degrade. The biodegradability of the resulting edible film can be determined

by burying the sample in compost soil which has more microorganisms than ordinary soil. The effect of adding carrageenan and sorbitol to % biodegradability can be seen in Figure 5.

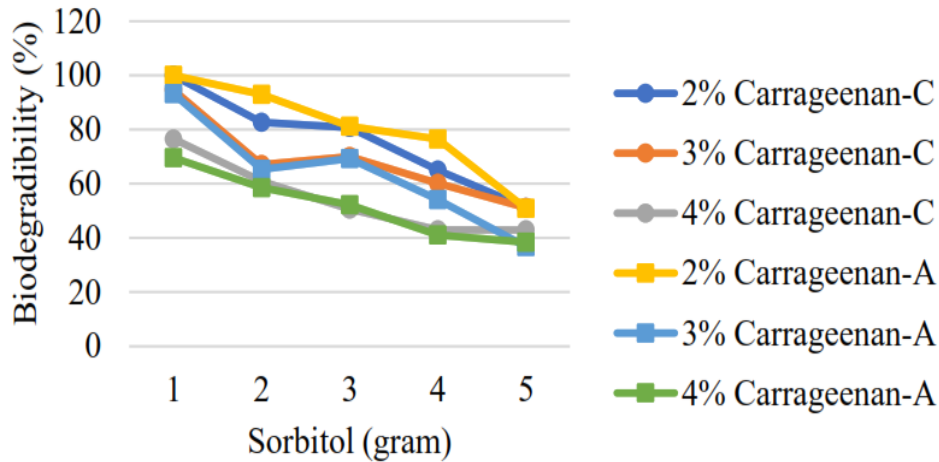


Figure 5. Graph of the relationship between the addition of carrageenan and sorbitol to the percentage of biodegradability in edible films.



Figure 6. Edible Film.

The results of the analysis show that the ability of biodegradability is affected by the composition of the edible film. In Figure 5, the edible film was completely degraded in variations A and C with a composition of 2% carrageenan and 1 gram of sorbitol. This is due to the addition of materials with low concentrations that reduce the elasticity and thickness of the edible film, which causes it to decompose easily in the soil. Meanwhile,

the edible film with a high concentration of ingredients in variation A with 4% carrageenan and 5 grams of sorbitol obtained low degradability. The cause of ease with which an edible film is degraded is due to the presence of hydroxyl and carbonyl groups which have hydrophilic properties so that water molecules come out of the plastic layer and provide space for microorganisms to enter [14].

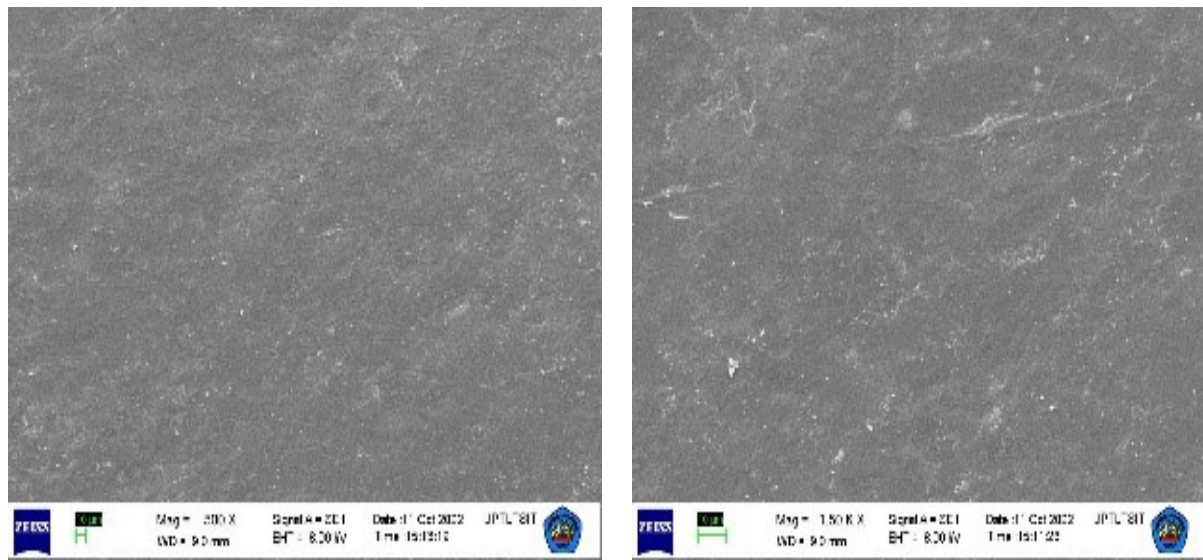


Figure 7. SEM results of edible films with a variation of 2% carrageenan concentration.

d. Surface morphology analysis with SEM (Scanning Electron Microscopy)

Edible films resulting from carrageenan variations were then analyzed by Scanning Electron Microscopy (SEM) to determine their surface morphology. This analysis was only carried out on edible films with a carrageenan variation of 2% because the resulting edible films did not match those shown in Figure 6. This was due to the comparison between carrageenan and sorbitol plasticizers added in small amounts. Where the ratio of the addition of sorbitol affects the formation of edible films. Sorbitol plasticizers can form good biopolymers and affect the film's physical properties.

Figure 7 shows a surface with a slightly porous structure and several cracks. This is due to the small addition of plasticizers in making edible films. Baldwin *et al.*, [15], stated that the addition of plasticizers needs to be done on films or coatings to avoid the presence of pores (cavities) and cracks. Thomazine *et al.*, [16], added that comparisons of carrageenan films with films containing several additives (plasticizers, surfactants) allow understanding and correlation of changes in surface properties with the properties of the additives used and

influence the orientation of the surface polymer chains during processing. Film formation [7].

Conclusions

Based on the results of this study, it can be concluded that glucomannan from local porang tubers can be made into edible films with environmentally friendly properties. However, a modification is needed to manufacture porang flour to reduce its oxalate content. The best treatment was obtained from the modified lime immersion and mocaf starter fermentation which reduced calcium oxalate levels and produced glucomannan yields close to the control treatment. Edible films made from treatment C showed that the combination of the addition of carrageenan and sorbitol plasticizers produced edible films with different characteristics. Thickness analysis of variation C 3% carrageenan and 4 g sorbitol, water resistance analysis, namely variation C 2% carrageenan and 5 g sorbitol and biodegradable analysis, namely variation A and C 2% carrageenan and 1 g sorbitol. Further research is still needed to get a plasticizer combination with a suitable combination.

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