Development and Characterization of Alginate Based Biocomposite Films Reinforced by Gracilaria Powder

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Abstract. The edible film is a thin sheet that functions as a coating or packaging material on foods that may be eaten simultaneously as packaged products. The ingredient that is often used is alginate made from seaweed. The advantage of using seaweed as a bioplastic material is that it can be produced in a large quantity, low price, and is non-toxic; it can also produce bioplastic that resembles conventional plastic. The experimental method with two components of Completely Randomized Factorial Design was employed in this study (CRFD), the concentration of Gracilaria seaweed powder with four levels (0%, 0.5%, 1%, 1.5%, and 1%) and concentration of alginate with three levels (1%, 1.25%, dan 1.5%). The result showed that the concentration of seaweed powder and concentration of alginate that used in the making of the edible film had a significant effect on the quality of the edible film on all parameters, which is thickness, moisture content, tensile strength, elongation, water vapor transmission rate, water-solubility, color, opacity and surface morphology. There is an interaction between the concentration of seaweed powder and concentration of alginate at the significant effect on the quality of the edible film, which is a concentration of seaweed powder at the level of 1.5% and concentration of alginate at the level of 1.5%. The film has a thickness of 0.25mm, moisture content of 6.94%, tensile strength of 54.29 Mpa, elongation of 3.26%, the water solubility of 64.41%, water vapor transmission rate of 3242 g/cm²/24 h, and opacity of 81.7%.

1. Introduction

Food can be packaged in various ways, including edible film, because it is readily decomposed by microorganisms and has environmentally friendly (biodegradable) properties. Based on Japanese industrial standards, The edible film is a thin coating that might be 0.25 mm thick, which are made of edible materials and can be coated with food (coating) or positioned between food components (film), which functions as an impediment to mass transfer [1]. Edible films from polysaccharides are usually made of starch, alginate, cellulose, carrageenan, or pectin. They have strong, dense, thick properties and good adhesion to protect the material from being packaged to be used as edible film [2].

Alginate is a primary metabolite of important hydrocolloid compounds widely used by the food and non-food industries. Alginate is also a natural polysaccharide that is viscous and soluble in water. The content of polysaccharides in Sargassum sp. is alginate, which reaches 40% of the total dry weight. Alginate in brown seaweed is obtained through the extraction process and functional group analysis [3]. Alginate has the potential as a basic ingredient for making edible films because the film formed
will be strong, easily decomposed, non-toxic, and able to form gels with the addition of Ca\(^{2+}\) ions so that alginate can be used as a starting ingredient for edible films [4].

Gracilaria sp. is a red seaweed that can be found in Indonesian waters. Seaweed resistant to freshwater may even exist in brackish water, which is why it is commonly seen on the coasts of huge islands. Gracilaria sp. generally contains agar (agarophytes) as a result of their primary metabolism. In addition, gracilaria can form a gel that can be used as a base for edible films.

Previous studies on edible films made from various natural polymers, such as alginate-gluten-beeswax composite, have been done [5], the effect of glycerol on edible films made from a whey-agar mixture [6], and the ratio of alginate/kappa-carrageenan on cross-linking composite films [5] [7]. In this study, the concentrations of Gracilaria powder and alginate were made to vary to know the effect of Gracilaria powder and alginate on the quality of the edible film. It is hoped that seaweed powder will get an edible film that can produce a good quality.

2. Materials and Method

2.1. Material
The ingredients used in the production of edible films are seaweed gracilaria powder (8.30% moisture content, gel strength 95.46 g/cm\(^2\)), alginate (6.89% water content, 30 cps viscosity), glycerol (Technical grade), and aquades.

2.2. Method
The method for making alginate based biocomposite film in this study refers to [8]. The method is using seaweed powder gracilaria and alginate. Gracilaria powder used to manufacture edible films consisted of four concentrations of 0; 0.5; 1; and 1.5% and added alginate with three concentrations of 1; 1.25; 1.5%, and 1 ml of glycerol. The manufacturing process consists of several stages, which include weighing the material, dissolving, heating to 100 °C, plasticizer (glycerol), pouring the solution into an acrylic plate with a size of 16x16 cm as much as 100 ml, and holding it at a controlled temperature for 24 hours. The analysis carried out were thickness, moisture content, tensile strength, percent elongation, water vapor transmission rate/WVTR, water solubility, scanning electron microscope, opacity, and color.

2.3. Data Analysis
The data analysis technique in this study was a two-factor Factorial Completely Randomized Design (CRFD). The CRFD used includes factor A (alginate) and factor B (concentration of seaweed powder), with two replications.

3. Result and Discussion

3.1. Thickness
Because the thickness of alginate based biocomposite film substantially impacts the physical properties, it is a crucial parameter in determining the practicality of edible film as food packaging. Several physical test characteristics, including as tensile strength, elongation, water vapor transmission rate and solubility, can be affected by the thickness of the edible coating. The average thickness of alginate based biocomposite film reinforced by Gracilaria powder ranged from 0.14±0.001–0.18±0.001 mm. The graph of the alginate based biocomposite film thickness can be seen in Figure 1.
The concentration of Gracilaria powder had a very significant effect on the thickness of the alginate based biocomposite film with the concentrations of 0% and 1.5% Gracilaria powder, while the concentrations of 0.5% and 1% gave an effect but were not significant on the thickness. The higher the concentration of seaweed powder added, the higher the thickness produced. This is presumably because the increasing concentration added will increase the distance between the polymeric gracilaria powder molecules to increase the thickness of the edible film. An increase in Gracilaria powder concentration will result in a rise in the polymer that makes up the edible film matrix, as well as an increase in total dissolved solids in the edible film, leading the edible film thickness to increase [11,12].

The amount of alginate in the edible film had a substantial impact on its thickness. The thickness created is proportional to the amount of alginate supplied. This is likely due to the fact that as the concentration is increased, the amount of dissolved solids in the edible film increases. The interaction between the components that make up the edible film, as well as the specific nature of colloid compounds as thickeners and suspenders, contribute to the growth in the thickness of the edible film. [13]. There was no interaction between Gracilaria powder and alginate on the thickness of the edible film.

Alginate, gluten, and beeswax composite edible films produced thicknesses ranged from 0.07-0.11 mm [14], and edible films with glycerol as plasticizer ranged from 0.03-0.08 mm [9]. Compared with the research above, the edible film in this study did not differ much in the range of 0.06-0.26 mm. The maximum film thickness value, according to the Japanese Industrial Standard (1975), is 0.25 mm. The alginate based biocomposite film reinforced by Gracilaria powder produced in this study meets the Japanese Industrial Standard's quality standards (1975).

3.2. Moisture content

The amount of moisture in the edible film plays a significant effont in the stability of the coated product. Low water content in its application as primary packaging does not contribute water to the development impacting product damage and decreasing shelf life [9].

The moisture content of alginate based biocomposite film reinforced by Gracilaria powder ranged from 12.29±4.80% – 23.08±2.01%. Figure 2 shows the moisture content of the biocomposite film as a graph.
Figure 2. Moisture Content of Alginate Based Biocomposite Films Reinforced by Gracilaria Powder

The moisture content of the edible film was significantly impacted by the concentration of alginate and seaweed Gracilaria powder. The lower the water content produced, the higher the concentration of alginate and seaweed Gracilaria powder added. This is because the insoluble solids in the edible film will rise as the concentration of the additional ingredient increases. Because alginate absorbs water, the density of the edible film will grow as the concentration is increased [10].

The phenomenon of a decrease in water content with an increase in the concentration of basic ingredients in the manufacture of edible films occurs because Gracilaria powder and alginate as basic ingredients carry solids dissolved in the solution for making edible films, causing hydrogen bonds to form between edible film constituent molecules. As a result, the edible film's free water content was lowered [9]. The edible film's moisture content is expected to be low to reduce sample damage and extend shelf life.

The moisture content of edible film made from alginate, gluten, and beeswax ranged from 21.95-24.63% [5], and edible film carrageenan with glycerol as plasticizer ranged from 17.14-20.86% [9].

3.3. Tensile Strength

One of the most crucial mechanical characteristics of edible films is tensile strength. Because it relates to the edible film's ability to protect the product with which it is coated. Food product packaging necessitates the use of edible films with high tensile strength, which aims to protect foodstuffs during handling, transportation and marketing [15].

Tensile strength of alginate based biocomposite film reinforced by Gracilaria powder ranged from 17.56±8.55 – 52.73±2.05 Mpa. The tensile strength graph can be seen in Figure 3.
Figure 3. Tensile Strength of Alginate Based Biocomposite Films Reinforced by Gracilaria Powder

The concentration of Gracilaria powder had a very significant effect on the tensile strength of edible films with the concentrations of Gracilaria powder 1% and 1.5%, while at concentrations of 0% and 0.5% gave a different, however, there is no discernible effect on the tensile strength of edible films. The tensile strength obtained increases as the concentration of grass powder increases. This is presumably because the increasing concentration added will increase the strength of the edible film matrix. This is by the statement of [16] that the general nature of cellulose has high tensile strength. The higher the concentration of material added in the manufacture of edible films, the stronger the film matrix. The force required to decide the edible film is also more significant [17].

Tensile strength of edible films as a function of alginate content varies significantly. The higher the alginate concentration, the greater the tensile strength. This is most likely due to the fact that increasing the concentration will increase the dissolved solids in the edible film tissue. Gracilaria powder and alginate had no effect on the tensile strength of edible films. Because the increase in alginate concentration on the edible film had no significant effect on the tensile strength value of the resulting edible film, the best tensile strength was at a concentration of 1.5% Gracilaria powder and 1% alginate.

The study results [5] showed that edible films based from alginate with beeswax and gluten addition produced tensile strength values ranging from 13.41-34.87 Mpa. Edible film carrageenan with glycerol as plasticizer has a tensile strength ranged from 4.17-6.66 Mpa [9]. Compared with the research above, the edible film in this study did not differ much in the range of 2.83-43.13 Mpa. The edible film's minimum tensile strength value is 3.92 Mpa, according to Japanese Industrial Standard. As a result, the biocomposite film produced in this study meets the Japanese Industrial Standard's quality standards.

3.4. Elongation

Elongation is the percentage increase in the length of the film when it is dragged till it tears. The percent elongation of alginate based biocomposite film reinforced by Gracilaria powder ranged from 5.12±2.10–23.38±0.44%. Figure 4 shows the elongation of the edible film as a graph.
The concentration of Gracilaria powder had a very significant effect on the elongation of the edible film with all concentrations of Gracilaria powder. The lower the % elongation, the higher the concentration of seaweed powder. This is likely due to the Gracilaria powder molecules forming a stronger film matrix, causing the film to become more inelastic or brittle, and therefore the elongation to decrease [18].

The elongation of the edible film was unaffected by alginate concentrations of 1.25% and 1.5%. While a concentration of 1% resulted in a substantial variation in the edible film's elongation. According to the findings, the smaller the percentage of elongation produced, the higher the alginate concentration. This is because the dissolved solids in the edible film tissue will grow as the concentration is increased. On the percent elongation of the edible film, there was no interaction between Gracilaria powder and alginate.

Alginate can bind water in larger quantities to produce a gel matrix that can increase the elongation of the edible film [19]. In contrast, the addition of Gracilaria powder elongation will be lower. The cellulose content will increase after being boiled at 100°C for 5 minutes [20]. Boiling causes swelling of the cell walls, resulting in the release of starch and lipids. So that it will increase the strength of the edible film matrix, which causes the elongation of the edible film to be decreased.

Edible films made from alginate composites, gluten, and beeswax produced a percentage of elongation ranging from 1–2.5% [19]. According to the Japanese Industrial Standard, the minimum percent elongation value is 5%. Thus, the edible film produced in this study is by the quality standards set by the Japanese Industrial Standard. The edible film with a low percentage of elongation indicates that the film is stiff, so it breaks easily and is not elastic [21].

3.5. Water Vapor Transmission Rate/WVTR
The WVTR is the amount of water vapor that passes through the film's surface per area [25]. The edible film water vapor transmission rate range from 3275.73±63.7–3933.57±108.4 g/m².24h. The graph of the WVTR of the edible film can be seen in Figure 5.
The concentration of Gracilaria powder has a significant effect on the water vapor transmission rate of the edible film. The higher the concentration of seaweed powder added, the lower the water vapor transmission rate produced; this makes the edible film better, inhibiting water vapor transmission rate. The best water vapor transmission rate was at a concentration of 1.5% Gracilaria powder but based on the standard JIS edible film in this study; it did not meet the standard; based on JIS, the maximum value for the water vapor transmission rate of the edible film was 10 (g/m2·day).

The results of the study showed that edible films are made from alginate composites. Gluten and beeswax produced water vapor transmission rate values ranging from 154.34–284.40 g/m2/24 hours [5]. When compared [5] the edible film in this study differed greatly in the range of 3275.73–3874.53 g/m2/24 hours. However, when compared to research [11], it has a lower water vapor transmission rate. According to JIS (1975), the maximum water vapor transmission rate is 10 g/m2/24 hours, so the edible film produced in this study is not by the quality standards set by JIS.

3.6. Water solubility
The solubility of edible films is measured in terms of their ability to dissolve and retain water in water. Because it is connected to the capacity of edible films to have water, solubility is an important physical attribute of edible films [22].

The average solubility value of alginate based biocomposite film reinforced by Gracilaria powder ranges from 65.05±4.41–90.25±6.99. The graph of the solubility of the edible film can be seen in Figure 6.
The concentration of Gracilaria powder has a significant effect on the solubility of the edible film, with the concentration of Gracilaria powder 1% and 1.5%. Meanwhile, the concentrations of 0% and 0.5% had no significant effect on the solubility of the edible film. The higher the concentration of seaweed powder, the lower the water solubility of the edible film. This is because the increasing concentration of Gracilaria powder will increase the number of intermolecular bonds [2], besides the content of Gracilaria powder generally consists of cellulose which is not soluble in water.

There is an interaction between Gracilaria powder and alginate which tends to have a significantly different effect on the solubility of edible films. The higher the concentration of Gracilaria powder and alginate added, the lower the resulting solubility.

Edible film carrageenan with glycerol as plasticizer has water solubility ranged from 60.51-74.20% [23]. When compared with the data in this study, it has a lower solubility, which is between 71.03-97.23%. The high solubility value causes the edible film to be easily soluble in water and reduces its ability to hold water.

Because they dissolve easily when swallowed, edible films with a high solubility are ideal for use in ready-to-eat food products [15]. The film's biodegradability is further influenced by its high solubility value. Low solubility value, on the other hand, is one of the most significant requirements for edible films, particularly for use as food packaging, which has a high water content and activity [24].

3.7. Color

The color of the edible film is one of the parameters that show the physical appearance of the edible film. The brighter the color, the better edible film produced.

| Warna       | Kode |
|-------------|------|
|             | A0B1 | A0B2 | A0B3 | A1B1 | A1B2 | A1B3 | A2B1 | A2B2 | A2B3 | A3B1 | A3B2 | A3B3 |
| L*          | 90.69 | 91.09 | 91.02 | 86.96 | 87.4 | 87.44 | 84.58 | 85.58 | 85.19 | 82.47 | 81.03 | 80.64 |
| a*          | -1.02 | -1.03 | -1.05 | -1.07 | -1   | -1.03 | -0.65 | -0.79 | -0.77 | -0.12 | 0.18  | 0.4  |
| b*          | 3.15  | 3.16  | 3.36  | 15.25 | 12.78 | 13.3  | 20.96 | 17.07 | 18.1  | 23.01 | 24.63 | 24.25 |

Table 1. Colour value of L*, a*, b* of Alginate Based Biocomposite Films Reinforced by Gracilaria Powder

Table 1 shows the results of different colors from different concentrations. The average color difference between concentrations was obtained through the formula $E = (\Delta L^2 + a^2 + b^2)^{0.5}$. The addition of Gracilaria powder has a $dE^*$ value of color differences range from 3.53±0.4913 – 24.83±1.16. The addition of gracilaria powder and alginate used significantly affected the color of the edible film produced. The addition of gracilaria powder and alginate resulted in an increase in the color value of the edible film. The increasing color value resulted in the appearance of the edible film getting darker following the color of the material from the yellow gracilaria powder.

The alginate concentration had a very significant effect on the color of the edible film with alginate concentrations of 1.25% and 1.5%, while it was not significantly different at a concentration of 1%; the higher the concentration of alginate added, the resulting color does not affect the color of the edible film. This is thought to be influenced by the color of the alginate base material, which is transparent white when it has become an edible film.

There was no interaction between gracilaria powder and alginate which had no significant effect on the color of the edible film. The results above stated that the higher the concentration of gracilaria powder and alginate added, the more concentrated the color produced following the color of the material from the golden yellow gracilaria powder.
In the study of edible film with a mixture of whey and agar by [26] edible film produced by treatment with glycerol concentration has a color value of L* (87.23–88.89), color a* (-0.215 – (-0.431), and color b* ( 3,464 – 3,474). The results of the study [27] using edible film with a concentration of glycerol and apple pulp peel extract have a color value of L (60.21 – 62.43). This result is different from the study results [28] showed that the edible film from breadfruit-chitosan starch polyblend had a value of L = 80.49. a = 2.29. b = -12.7. with a pale gray starch color which showed the characteristics of bright color and a bluish-red color when viewed from the a and b values. When compared with the three studies above, the edible film produced in this study has a color that tends to be darker where the color of the edible film depends on the color of the type of base material used where the basic color of seaweed powder is Gracilaria has a yellow color. Alginate is ivory white while glycerol is colorless or clear.

3.8. Opacity

Edible film opacity is one of the parameters that show the transparency of edible films. The opacity of the sample is an indication of how much light can penetrate the sample. The higher the opacity value, the lower the amount of light that can penetrate the sample. The average opacity of edible films can be seen in Table 2.

| Alginate  | Gracilaria powder |
|-----------|-------------------|
|           | 0% | 0.50% | 1% | 1.50% |
| 1%        | 181.63 | 174.88 | 166.285 | 164.055 |
| 1.25%     | 183.06 | 174.355 | 170.545 | 162.195 |
| 1.50%     | 181.75 | 174.45 | 168.77  | 163.405 |

From the results, it was found that the addition of the concentration of Gracilaria powder had an opacity value ranging from 81.60±0.4 - 91.0±0.39.

The addition of Gracilaria powder and different alginites resulted in a decrease in the opacity value of the edible film, the lower the opacity value resulted in the appearance of the edible film becoming darker/not translucent. The addition of the concentration of Gracilaria powder and the interaction with the alginate used significantly affected the opacity of the edible film produced. Meanwhile, the addition of alginate concentration did not significantly affect the opacity value of the resulting edible film.

The concentration of Gracilaria powder had a very significant effect on the opacity of the edible film with concentrations of 0%, 0.5%, 1%, and 1.5% of Gracilaria powder. The results above stated that the higher the concentration of seaweed powder added, the lower the resulting opacity.

The interaction between Gracilaria powder and alginate has an effect that tends to be not significantly different on the opacity of the edible film.

According to research results [21], edible film is based on alginate with garlic extract. red ginger and temulawak produced an opacity value of 22.63±1.47-6.03±0.43%. Research results (Mulyadi et al., 2016) edible film with the addition of beluntas leaf extract has an opacity of 55 - 65.15%. Research results [18] in [21] edible film with the addition of microalgae extract has an opacity value of 40.2 – 77.4%. When compared with the three previous studies, it can be said that the opacity results obtained from the addition of Gracilaria powder and alginate used resulted in higher values 81.60±0.4 - 91.0±0.39%.
3.9. **Surface Morphology**

The surface morphology of the alginate based biocomposite film with the addition of Gracilaria powder and alginate can be seen in Figure 7.

![Morphology Images](a) (b) (c) (d) (e) (f) (g) (h) (i) (j) (k) (l)

**Figure 1.** Surface morphology of alginate based biocomposite film up to 500x magnification

Description a: A0B1. b: A0B2. c: A0B3. d: A1B1. e: A1B2. f: A1B3. g: A2B1. h: A2B2. i: A2B3. A: A3B1. h: A3B2. i: A3B3

From Figure 7, it can be seen that the molecules of alginate, which are the ingredients for making edible films, look less homogeneous and have an even texture (A0B1-A0B3). In contrast, the surface of the edible film added with Gracilaria powder looks rounded texture (A1B1-A3B3). It is also seen that the distance between the particles is not tight. It is feared that it will affect the quality of the edible film in restraining the rate of water vapor transmission. Based on the parameters of the physical test and chemical test where the best concentration was selected with the addition of 1.5% Gracilaria powder and 1.5% alginate, the morphological characteristics of the edible film were quite clear, where the surface of the edible film was not homogeneous when viewed from the scanning electron
microscope. This is in accordance with [28] with less dense structure or cracks of these fibers causing more water to be absorbed.

4. Conclusion
The concentration of Gracilaria powder in the alginate based biocomposite film has a significantly affects on their quality. Parameters affected by the concentration of Gracilaria powder were thickness, moisture content, tensile strength, percent elongation, WVTR, water solubility, opacity, and color. Parameters influenced by alginate concentration were thickness, moisture content, tensile strength, elongation, and color. There is a different interaction between the concentration of Gracilaria powder and alginate, which significantly affects the quality of the alginate based biocomposite film. The affected parameters are water content, thickness, tensile strength, percent elongation, solubility, opacity, and color. Based on the number of the best quality parameters, the alginate based biocomposite film was chosen with a concentration of 1.5% Gracilaria powder and 1% alginate, which has a thickness of 0.19 mm, moisture content of 16.24%, tensile strength of 50.4 Mpa, elongation of 22.869, solubility 64.41%, WVTR of 3161g/cm²/24 h, opacity of 81.7%, and surface texture that is less homogeneous.

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