The effect of organic powdered cottonii concentration and types of plasticizers on the characteristics of edible film

D Fransiska¹, Giyatmi², J Basmal¹, E Susanti²

¹ Research Centre of Marine and Fisheries Food Processing and Biotechnology, Jl. KS Tubun Petamburan VI, Slipi, Jakarta Pusat, Indonesia
² Faculty of Food Technology and Health, Sahid University, Jl. Prof. DR. Soepomo, Tebet, Jakarta Selatan, Indonesia
*e-mail: dinanomo@gmail.com

Abstract. Edible film is a thin layer that can be used as an alternative packaging or food coating that can be eaten along with the products labelled with. One of the materials used to make an edible film is Organic Powdered Cottonii (OPC), a product containing carrageenan and produced from Eucheuma cottonii seaweed. This research used Completely Randomized Factorial Design (RALF) method with two factors, namely PC concentration (A) with four levels (1 %; 1.5 %; 2 %; 2.5 %) and types of plasticizer (B) with three levels (Glycerol; PEG; Sorbitol) with three replications. The results showed that PC concentration had a significant effect (sign < α (0.05)) on the parameters of moisture content, thickness, tensile strength, elongation, and water solubility. Different types of plasticizers have significant effects (sign< α (0.05)) on the moisture content, thickness, tensile strength, elongation, water solubility, and water vapor transmission rate (WVTR). There is an interaction between PC concentration and type of plasticizer in PC concentration of 2.5 % and the type is sorbitol. The results of the edible film yield moisture content value of 8.30 %, thickness value of 0.09 mm, tensile strength of 12.08 MPa, elongation value of 1.67 %, water solubility value of 49.99 %, and water vapor transmission rate of 3.30 g/cm²/24 hours.

Keywords: Edible film; Organic Powdered Cottonii (OPC); glycerol; PEG; sorbitol

1. Introduction

Edible film is one alternative packaging that can be applied to food due to its biodegradable nature. It is a thin layer that functions as a food packaging or coating that can be eaten together at once with the food wrapped in it [1]. Edible film is generally composed of biopolymers of agricultural products that are easily found and demolished biologically (biodegradable) such as polysaccharides, fats or a combination of the two.

Research on edible film has been carried out with a variety of basic materials, such as edible film with carrageenan and chitosan [2], semi-refined Carrageenan flour and tapioca flour [1], carrageenan and glycerol [3], sugar palm fruit and a variety of plasticizers [4], or organic powdered cottonii (OPC) base material [5]. OPC is a carrageenan product obtained from seaweed with the type of Eucheuma cottonii (red seaweed) by immersion treatment using alkaline solution of potassium hydroxide (KOH). OPC is yellowish-white, flour, and can form gel that is important in the food and drug industry, as a stabilizer, thickener and emulsifier.
Making edible film using OPC will produce film with low elasticity value [5]. Therefore, it is necessary to add a plasticizer which plays a role to improve the elasticity and flexibility of the film. Commonly used plasticizers are glycerol, sorbitol, and polyethylene glycol (PEG). Edible film with sorbitol plasticizer has greater tensile strength and water vapour transmission rates compared to those with glycerol plasticizer, which are 8.68 MPa and 5.34 g/cm²/24 h, respectively [5]. Edible film with sorbitol plasticizer has greater tensile strength and water vapour transmission rate compared to those with PEG plasticizer, which are 2.8 MPa and 4.34 g/cm²/24 h [4]. The solubility value of edible film with sorbitol plasticizer is smaller compared to glycerol plasticizer which is approximately 64.75 % [5] but when compared to PEG plasticizer, edible film with sorbitol plasticizer has a greater value of 62.35 % [4]. In this study, various concentrations of OPC were used in order to get a good formulation of making edible film and the effect of different types of plasticizers to obtain better edible film characteristics.

2. Methodology

2.1. Materials
The materials used in the process of making edible film are OPC, plasticizers (Glycerol, PEG, and sorbitol), and distilled water. OPC raw materials used were obtained from the Research Centre of Marine and Fisheries Food Processing and Biotechnology, Slipi, Central Jakarta. Glycerol and Sorbitol were obtained from PT. Geochem Mitra Perkasa and PEG was from PT. Brataco.

2.2. Method

2.2.1. Film preparation and characterization
The preparation of OPC edible film refers to the method used by [5], which has been modified at a concentration of base material and type of plasticizer. OPC raw materials (1, 1.5, 2, and 2.5 %) were mixed with water in a beaker glass and stirred using a magnetic stirrer rotating on a turned-off hotplate. After homogenous mixing, the hotplate was turned on until the solution reached a temperature of 85 ºC. Then plasticizer (glycerol or PEG or sorbitol) was added according to the formulation while stirring and mixing at 85 ºC. The film solution was then poured into 16 x 16 cm² acrylic mould. The moulds were allowed to stand at a room temperature (27 ºC) for 24 h. Later than, the film was dried in an oven at 40 ºC for 24 h. Thereafter, it was out from the oven, taken off from the moulds, put it in a plastic clip and stored in a desiccator at a room temperature.

2.2.2. Characteristic
Parameter on the characteristics of raw materials includes moisture content by the oven (SNI-01-2354.2-2015), ash content (SNI-01-2354.1-2006), acid insoluble ash content (SNI-01-2354.1-2010), and sulfate content (Gravimetric Method). Meanwhile observation on edible film quality includes moisture content (SNI-01-2354.2-2015), thickness (Mitutoyo Micrometer) [3], tensile strength and elongation (TA XT analyzer), solubility [6], and water vapour transmission rate [7]. The tests were carried out in three replicates.

3. Results and Discussions

3.1. Characteristics of Base Materials
Chemical analysis was performed on OPC prior to processing to evaluate whether the used materials are suitable or not. The testing results of OPC materials are presented in Table 1.
Table 1. Testing results of organic powdered cottonii materials.

| Parameters                        | Value   |
|-----------------------------------|---------|
| Moisture content (%)              | 9.11±0.10 |
| Ash content (%)                   | 24.24±0.29 |
| Acid insoluble ash content (%)    | 0.52±0.41 |
| Sulphate content (%)              | 4.81±0.48 |

The moisture content in a material has a great impact on its shelf life [8] and it may affect the texture, storability, taste and appearance of a material. Moisture content in food indicates the resistance of the materials to microbial attack. The higher the moisture content in a material, the faster the material will be deteriorated. The value of moisture content from the test was 9.11±0.10 % where this indicates a lower value than the carrageenan moisture content standard issued by FAO [9], a maximum of 12 %. This low value of OPC moisture content is assumed to be caused by KOH ability in extracting and inhibiting the increase in water in the Eucheuma cottonii seaweed molecule where the higher KOH concentration used will result in the lower value of moisture content [10]. The higher KOH concentration during extraction will increase pH values so that the KOH ability in extracting seaweed is also greater and the moisture content would be lower [11]. The lower the value of the moisture content, the better the quality of the OPC.

Ash content analysis was carried out to determine the total mineral content in OPC. The ash content value of food shows the amount of minerals contained in that food [8]. The resulting ash content was 24.24 %. Increase of the ash content is due to the number of K⁺ cations that reacted with more OPC or vice versa. The high value of ash content in OPC flour could also be caused by most of the formed ash coming from the salt and minerals attached to the seaweed such as K, Mg, Ca, Na and ammonium galactose [10]. According to FAO standardization [9], the maximum ash content quality criteria are 15-40 %, so the OPC obtained in this study has met the quality criteria as they are within the specified limits.

Acid insoluble ash content is insoluble chloride salts which are mostly heavy metal salts and silica found in nature such as sand or stone [12]. Acid insoluble ash content is a criterion for determining the level of cleanliness in OPC processing. The value of acid insoluble ash content in this study is 0.52%. This low acid insoluble ash content showed that the OPC used in this study was less contaminated during the process of handling and processing raw materials [8]. According to FAO [9], the quality standard of carrageenan acid insoluble ash content is maximum 1 %, so it still meets the specified quality standard.

Sulphate content is a parameter used for various types of polysaccharides found in red algae. Sulphate content is closely related to the quality of the carrageenan produced, especially to the gel strength. The higher the sulphate content, the lower the gel strength and vice versa [13]. This is justified by the statement that the high sulphate content in carrageenan may inhibit the process of gel formation [13]. The results of sulphate content obtained in the test is 4.81 %, which is lower than the sulphate content standard set by FAO or FCC which is around 15-40 %. The low value of sulphate content of a carrageenan will increase its glassy nature [15].

3.2. Characteristics of edible film

3.2.1. Moisture content

Moisture content in OPC edible film is measured to determine the moisture content that remains in storage, as the water may affect the texture, storability, taste and appearance of the film. The average values of moisture content obtained are presented in Table 2.
In Table 2, the average moisture content of four concentrations ranges from 17.88±0.86 to 9.58±1.49 %, with the highest value of 17.88±0.86 % for 1 % OPC concentration and the lowest at 9.58±1.49 % for 2.5 % OPC concentration. Meanwhile, the average value of moisture content for different types of plasticizers ranges from 15.09±2.94 to 11.51±3.75 %, with the highest value of 15.09±2.94 % for glycerol plasticizer, and the lowest value of 11.51±3.75 % for sorbitol. The values of moisture content tend to decrease with the increasing OPC concentration used. This is in accordance with the statement that the increase of carrageenan concentration in making edible film tends to reduce the moisture content of the edible film produced [15]. The low polysaccharide concentration in the edible film forming solution allows the availability of more free water to participate in the polymerization reaction, thus resulting in an edible film with a relatively high moisture content value [16].

In each plasticizer, glycerol, PEG, and sorbitol have similar results that are the higher the OPC concentration used, the moisture content also tends to decrease. In addition, it can be seen that the PEG plasticizer has a higher value compared to other plasticizers. This is due to the more hydrophilic nature of PEG plasticizer so that the water binding ability is greater than glycerol or sorbitol. Sorbitol has a lower ability to bind water compared to other plasticizers; this probably causes the moisture content of edible film with sorbitol plasticizer lower [17].

The results of this study indicate that edible film using glycerol and PEG plasticizers has higher moisture content than sorbitol. This is in accordance with the finding of Khoiro [5] in his research with OPC which concluded that edible film with glycerol plasticizer has greater moisture content than that of sorbitol. PEG in edible film processing will produce greater moisture content compared to sorbitol [4]. The best edible film is the one which has the lowest moisture content [18]. The lowest moisture content obtained in this study is 11.51 % found in edible film with sorbitol. The low moisture content indicates the ability of edible film to protect the product longer.

### 3.2.2. Thickness

Film thickness is a parameter that affects the use of edible film in the formation of packaged products. The thickness values obtained from this study are presented in Table 3. Based on the data in Table 3, it is known that the average thickness of OPC edible film at four concentrations ranges from 0.05±0.00 to 0.08±0.01 mm, with the highest value at 2.5 % PC concentration of 0.08±0.01 mm, and the lowest at 1 % PC concentration of 0.05±0.00 mm. Whereas the average thickness value of OPC edible film on different types of plasticizers ranges from 0.07±0.01 to 0.06±0.01 mm, the highest value for glycerol and PEG plasticizers of 0.07±0.01 mm, and the lowest value of 0.06±0.01 mm.
The increase of OPC concentration used in making edible film will cause an increase in dissolved solids in the edible film forming solution, so that the thickness of the edible film will increase as well. Film thickness is influenced by material concentration, an increase in this concentration will cause an increase in the film thickness, because the higher the amount of material used in the film matrix, the higher the total solids [19]. Film thickness is mainly influenced by the concentration of dissolved solids in the film-forming solution. The types of plasticizers used namely glycerol, PEG, and sorbitol affected the thickness value of the OPC edible film produced. Based on Table 3, it is known that the average value of edible film thickness using PEG plasticizer is higher compared to other plasticizers. Edible film using PEG plasticizer had the highest thickness value and the lowest thickness was with sorbitol [4]. This difference is due to the plasticizer ability to absorb water and various resulting solids. The more water that is bound, the thicker the resulting edible film will be.

The thickness of OPC edible film obtained in this study is good as explained in Table 3 that the average thickness obtained is around 0.05-0.08 mm, so it is below the maximum standard of edible film thickness according to Japanese Industrial Standard that is 0.25 mm. The thickness results in this study have the same value with the research [5] which is around 0.05-0.06 mm and smaller than the research [1]) which has a thickness of 0.09 mm.

### 3.2.3. Tensile strength

In Table 4, it shows that the average tensile strength of OPC edible film at different concentrations ranges from 6.00±1.71 to 11.03±1.32 MPa, with the highest value of 11.03±1.32 MPa for 2.5 % OPC concentration, and the lowest of 6.00±1.71 MPa for 1 % OPC concentration. On the other side, the average value of tensile strength on the types of plasticizers ranges from 7.97±2.59 to 9.87±1.91 MPa, with the highest value of 9.87±1.91 MPa for sorbitol, and the lowest of 7.97±2.59 MPa for PEG. Carrageenan edible film that edible film tends to experience an increase in tensile strength along with the increased concentration of carrageenan added [20]. The higher the concentration of carrageenan added in making edible film, the stronger the film matrix will be.

OPC edible film that uses sorbitol tends to have greater average value than that of other plasticizers (Table 4). The tensile strength value of OPC edible film with sorbitol is better than glycerol [5]. The tensile strength of wheat-flour-based films with sorbitol plasticizer is better than using glycerol and PEG plasticizers [4]. This is probably due to the molecular structure of the sorbitol ring, which prevents insertion between chains, making it less effective in inhibiting interactions between proteins [21]. It was further stated that the low ability of sorbitol to bind water, thus limiting its ability to reduce the polymer chain hydrogen bonds compared to glycerol and PEG.

### Table 3. Analysis results on the thickness of organic powdered cottonii edible film

| PC Concentration | Glycerol  | PEG      | Sorbitol | Average |
|------------------|-----------|----------|----------|---------|
| 1.0              | 0.06±0.00 | 0.05±0.00| 0.05±0.00| 0.05±0.00|
| 1.5              | 0.07±0.01 | 0.06±0.01| 0.05±0.00| 0.06±0.01|
| 2.0              | 0.07±0.01 | 0.07±0.01| 0.07±0.00| 0.07±0.00|
| 2.5              | 0.08±0.00 | 0.00±0.00| 0.08±0.00| 0.08±0.01|
| Average          | 0.07±0.01 | 0.07±0.01| 0.06±0.01|         |
Table 4. Analysis results on the tensile strength of organic powdered cottonii edible Film

| OPC Concentration | Glycerol   | PEG     | Sorbitol | Average |
|-------------------|------------|---------|----------|---------|
| 1.0               | 6.47±0.14  | 4.10±0.32 | 7.43±0.43 | 6.00±1.71 |
| 1.5               | 7.76±0.66  | 8.85±0.93 | 9.86±0.45 | 8.82±1.05 |
| 2.0               | 9.61±0.67  | 9.36±1.10 | 10.12±0.78 | 9.70±0.39 |
| 2.5               | 11.46±0.65 | 9.55±0.13 | 12.08±0.74 | 11.03±1.32 |
| Average           | 8.83±2.18  | 7.97±2.59 | 9.87±1.91 |

Tensile strength is the maximum tensile force that a film can withstand. Too small tensile strength indicates that the edible film probably cannot be used as packaging, because its physical character is less strong and easily broken [22]. Different carrageenan concentrations will increase the interaction of carrageenan molecules and plasticizers in the film matrix and cause the formed film matrix to become sturdier and more compact, so cutting the film requires a greater force [23]. The tensile strength value obtained in this study was 12.08 %, smaller than research [1] at 39.42 %, but greater than the specified Japanese Industrial Standard at a minimum of 3.92 MPa. Therefore, the tensile strength of OPC edible film in this study meets the specified tensile strength standard.

3.2.4. Elongation

Elongation is the increase of the length of film material from the initial length at the time of extraction until it breaks. The elongation value (in %) indicates the ability of edible film to elongate. Generally, elongation value is inversely proportional to the tensile strength value. The average value of elongation of OPC edible film can be seen in Table 5. Based on the data in Table 5, the elongation value by OPC edible film with four PC concentrations ranges from 3.58±2.21 to 10.49±7.26 %, where the highest value at 1% PC concentration is 10.49±7.26 %, and the lowest at 2.5 % PC concentration is 3.58±2.21 %. Whereas the average value of the elongation of OPC edible film on on different types of plasticizers ranges from 2.70±1.67 % to 11.30±5.40 %, with the highest value on glycerol of 11.30±5.40 % and the lowest value on sorbitol of 2.70±1.67 %.

The test results of the edible film elongation with different concentrations indicate that the higher the carrageenan concentration, the elongation of carrageenan edible film will decrease [23]. The increase in OPC concentration used in making edible film will result in increase of tensile strength and decrease of elongation. The use of a greater OPC amount will make better ability to bind water so that it will produce gel that will increase the elongation of edible film. The research on protein films showed that increasing the film tensile strength breaks will be followed by a decrease in the elongation [24]. The use of glycerol plasticizer in this study has an average elongation value which tends to be greater than other types of plasticizers. These results are similar to Khoiro’s finding which suggests that the film elongation value with glycerol is higher than that with sorbitol [5]. The type of plasticizer used will affect the resulting elongation value.

The research results obtained on the elongation value of OPC edible film range from 1.67 to 18.77 %, where the value is still smaller compared to the results [5] in his research that is OPC edible film with a 1.5 % concentration is approximately 7.65-13.20 %, and the study of edible film with different types of plasticizers conducted by [4] also showed much greater elongation value compared to a study on OPC edible film conducted at 44.65 %.
Table 5. Analysis results on the elongation of organic powdered cottonii edible film

| PC Concentration | Glycerol | PEG    | Sorbitol | Average   |
|------------------|----------|--------|----------|-----------|
| 1.0              | 18.77±0.35    | 7.50±0.72 | 5.20±0.95 | 10.49±7.26 |
| 1.5              | 10.97±0.47    | 3.90±0.70 | 2.07±0.15 | 5.65±4.70  |
| 2.0              | 9.47±0.45     | 3.33±0.42 | 1.87±0.55 | 4.89±4.03  |
| 2.5              | 6.00±0.56     | 3.07±0.93 | 1.67±0.06 | 3.58±2.21  |
| Average          | 11.30±5.40    | 4.45±2.06 | 2.70±1.67 | 47.70±2.11 |

3.2.5. Solubility
The measurement of edible film solubility aims to determine the ability of edible film to dissolve in water and to hold water. The average solubility values of OPC edible film in this study are presented in Table 6.

In Table 6, it is known that the average solubility of OPC edible film at various concentrations ranges from 41.43±8.11 % to 47.10±3.26 %, where the highest value at 2.5 % PC concentration was 47.10±3.26 %, and the lowest value at 1 % concentration is 41.43±8.11 %. Whereas the average solubility by OPC edible film on different types of plasticizers ranges from 40.02±5.39 % to 47.70±2.11 %, where the highest value is glycerol plasticizer of 47.70±2.11 %, and the lowest value is PEG plasticizer of 40.02±5.39 %.

The higher the carrageenan concentration added, the greater the solubility will be as carrageenan and glycerol is hydrophilic [20]. A similar result is also suggested that solubility in water is an indication of hydrophilicity of hydrophilic carrageenan edible film [6]. Glycerol has the greatest solubility compared to other plasticizers. This is consistent with research that the solubility of OPC edible film with glycerol has greater value than that with sorbitol [5]. The increase in edible film solubility is due to hydrophilic nature of the plasticizer that increases its solubility due to the nature of glycerol as a plastic forming.

The best edible film in this study is the one with 2.5 % OPC concentration and glycerol because they have the highest solubility values. The application of a film is expected to be a worth-eating packaging, and then a high solubility is needed [18]. The highest solubility value of edible film in this study is 49.99±0.69 %, but the value is fairly low compared to the solubility of OPC edible film approximately 59.00 %. Research on carrageenan-based edible film [3] has a solubility value of 60.51 %, while the research [4] on edible film using various types of plasticizers had a relatively high solubility value of 62.35 %.

Table 6. Analysis results on the solubility of organic powdered cottonii edible film

| PC Concentration | Solubility (%) | Average |
|------------------|----------------|---------|
|                  | Glycerol       | PEG     | Sorbitol  |           |
| 1.0              | 45.10±0.91     | 32.13±0.47 | 47.05±0.64 | 41.43±8.11 |
| 1.5              | 47.03±0.98     | 40.99±0.67 | 47.45±0.76 | 45.16±3.61 |
| 2.0              | 48.67±0.62     | 43.37±0.25 | 47.62±0.31 | 46.55±2.81 |
| 2.5              | 49.99±0.69     | 43.57±0.38 | 47.75±0.53 | 47.10±3.26 |
| Average          | 47.70±2.11     | 40.02±5.39 | 47.47±0.30 |           |
3.2.6. Water vapour transmission rate

Water vapour transmission rate (WVTR) is the flow rate of water vapour through a unit area at a certain time and under certain conditions. The average value of WVTR of OPC edible film can be seen in Table 7. It shows that the average WVTR of OPC edible film to four OPC concentrations ranges from 3.54±0.32 to 3.70±0.52 g/cm²/24 h, where the highest is at 2 % OPC concentration of 3.70±0.52 g/cm²/24 h, and the lowest is at 2.5 % OPC concentration of 3.54±0.32 g/cm²/24 h. Whereas for different types of plasticizers, the average WVTR of OPC edible film ranges from 3.30±0.07 to 3.94±0.21 g/cm²/24 h, with the highest value on sorbitol of 3.94±0.21 g/cm²/24 h, and the lowest value on glycerol of 3.30±0.07 g/cm²/24 h.

In addition, of the three types of plasticizers, it is known that sorbitol tends to have the highest WVTR compared to other plasticizers. This is in line [5] where OPC-based edible film has the highest WVTR using sorbitol compared to glycerol. Sorbitol has lower ability to bind water compared to glycerol and PEG, resulting in high WVTR as the water vapour is released directly [14]. This is caused by differences in hygroscopic properties and in the chemical structure of plasticizers [4].

Edible film with base materials of Semi-refined Carrageenan and beeswax [18] have WVTR ranging from 2.18-2.58 g/cm²/24 h, smaller than the results obtained in this study which is around 3.30-3.94 g/cm²/24 h. However, the rate in this study is still smaller compared to the one studied by [20] with the basic ingredients of tilapia skin gelatine and carrageenan which is around 5.83-14.39 g/cm²/24 h. Low WVTR will inhibit the loss of water from products packaged with edible film, so that it inhibits the damage due to the activity of microorganisms. The maximum standard limit of WVTR set by Japanese Industrial Standard is 10 g/cm²/24 h, so that OPC edible film in this study is within the specified standard.

### Table 7. Analysis results on water vapour transmission rate of organic powdered cottonii edible film

| PC Concentration | Water vapour transmission rate (g/cm²/24 h) | Average |
|------------------|-------------------------------------------|---------|
|                  | Glycerol | PEG | Sorbitol |                   |
| 1.0%             | 3.37±0.18 | 3.54±0.89 | 3.73±0.07 | 3.55±0.18         |
| 1.5%             | 3.34±0.11 | 3.71±0.08 | 3.90±0.29 | 3.65±0.28         |
| 2.0%             | 3.21±0.08 | 3.66±0.17 | 4.24±0.04 | 3.70±0.52         |
| 2.5%             | 3.27±0.40 | 3.46±0.05 | 3.89±0.08 | 3.54±0.32         |
| Average          | 3.30±0.07 | 3.59±0.11 | 3.94±0.21 |                   |

4. Conclusion

The use of Organic Powdered Cottonii (OPC) significantly affected the characteristics of resulting edible film (moisture content, thickness, tensile strength, elongation, and solubility). The parameter that did not significantly affect OPC concentration was water vapour transmission rate. The best OPC concentration was 2.5 %. Various types of plasticizers significantly affected the characteristics of edible film (moisture content, thickness, tensile strength, elongation, solubility, and water vapour transmission rate). The best type of plasticizer was sorbitol. There was interaction between OPC concentration with the type of plasticizer in OPC edible film and significantly affected the characteristics of OPC edible film. The best interaction that affected the characteristics of OPC edible film was the one with 2.5 % OPC concentration and sorbitol, with a moisture value of 8.30 %, thickness of 0.09 mm, tensile strength of 12.08 MPa, elongation of 1.67 %, and solubility of 49.99 % and water vapour transmission rate of 3.30 g/cm²/24 h.
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