Effects of the Type and Concentration of Starch on the Quality of Alginate-based Edible Film

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Abstract: Edible film is a thin layer as an alternative packaging or food coatings that can be consumed. Ingredients that can be used for manufacturing edible film are alginate and starch. Alginate is normally extracted from brown seaweed and starch. The advantages of using seaweed and starch as edible film ingredients are low cost and non-toxic. This study employed a Completely Randomized Factorial Design with two factors of, namely the type of starch (A1 = canna starch, A2 = mocaf starch, A3 = breadfruit starch) and concentration of starch (B1 = 0 %, B2 = 0,1%, and B3 = 0,3%, and B4 = 0,5%). Experiment was conducted three replications. The results showed that the different type of starch had a significant effect ($\alpha<0.05$) on the thickness, tensile strength, elongation, and water solubility. The concentration of starch had significant effects ($\alpha<0.05$) on the moisture content, thickness, tensile strength, elongation, water solubility, and water vapor transmission rate (WVTR). The best alginate-based edible film was processed using 0.5% mocaf. Mechanical and physical properties of that alginate-based edible film were 9,91% moisture content, 5,21 Mpa tensile strength, 28,90% elongation, 97,93% water solubility, and 1709,287 g/cm^2/24 hour water vapor transmission rate. Morphological analysis using SEM (Scanning Electron Microscopy) revealed that the surface of alginate-based edible film was quite smooth and dense but a few starch granules was still found.

1 INTRODUCTION

Edible film is a thin layer that can be used as a food packaging or coating which can be consumed together with the packaged products. Edible film is employed by wrapping, brushing or spraying to provide selective control of the movement of gas, water vapor, and solutes as well as protection against mechanical damage (Sitompul and Zubaidah, 2017).

The main components for producing edible film can be grouped into three categories, namely hydrocolloids, lipids, and composites. Some types of hydrocolloids that can be used for edible film materials include gelatine, alginate, starch and others (Milani and Maleki, 2012).

Alginate is a natural polymer which is the main component of brown algae (*Phaeophyceae* sp) and an important compound in the cell walls of algae species belonging to the *Phaeophyceae* class. Edible films from alginate have high hydrophilic properties. The hydrophilic nature of alginates promotes edible films made from alginate to absorb water molecules (Anward, et al, 2013). According to Murdinah, et al. (2007), alginate has a potential to be processed into edible film, because alginate is rigid, edible and renewable nature. Producing edible film from alginate is one of the efforts to increase the utilization of seaweed.

Starches are composed of amylose and amylopectin molecules. Amylose is a fraction that plays a role in gel formation. Starch has properties that are easy to form gelatinization making it easily applied as a forming material and can produce good plastic (Afriyah, et al, 2015). Breadfruit flour, Modified Cassava Flour (mocaf) and canna flour are sources of starch that have not been widely used in the development of edible films. Breadfruit flour have amylose and amylopectin contents of 23.95% and 76.05% respectively (Afriyah, 2015). According to Diniyah, et al (2018), starch content of mocaf is

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relatively high, i.e. 85-87%. Whereas canna flour contain starch with amylose and amylopectin levels of 24.44% and 78.86% correspondingly (Santoso, et al, 2007).

Based on previous study on edible films with various natural polymers such as edible film consisting of alginate-gluten-beeswax (Murdinah, et al, 2007), the results revealed that edible films with the best composites of alginate composites, gluten and beeswax (1: 1: 2) did not affect the moisture content, thickness, and tensile strength but significantly affected the elongation and water vapour transmission rate of the film.

This study developed edible films made from natural polymers including alginate and three different types of starch, i.e. canna starch, mocaf starch and breadfruit starch with sorbitol as plasticizer.

2 MATERIAL AND METHODS

2.1 Material

The main ingredients used for developing alginate-based edible films were alginate, canna starch, mocaf starch and breadfruit starch. Alginate was extracted from brown seaweed (*Sargassum* sp) purchased from Gunung Kidul. Other ingredients were distilled water, sorbitol and beeswax. The characteristics of alginate and starches are shown in Table 1.

Table 1: Characteristics of Alginate and Starches.

Parameters of Analyses	Alginate	Canna Starch	Mocaf Starch	Breadfruit Starch
Moisture (%)	9.99±0.48	15.00±0.13	9.78±1.13	11.13±0.09
Ash (%)	23.27±0.17	0.25±0.14	1.69±0.04	2.79±0.01
Viscosity (cP)	30.83±4.73	15.83±1.44	18.33±1.44	14.00±1.32
Brightness	33.77±0.80	74.53±0.23	84.87±0.23	70.73±0.12

Moisture content of alginate was in accordance with the quality standards of Kennedy and Bradshaw (1987), which was <13%. The moisture content of mocaf and breadfruit starch met the Indonesian National Standard (SNI) of mocaf flour which was <13%, while the moisture content of canna starch was higher than SNI quality standards. The ash contents of alginate and starch met the quality standards of Kennedy and Bradshaw (1987), i.e. <23%. Viscosity value of alginate produced in this study was in accordance with the quality standards of Kennedy and Bradshaw (1987) which is 10 - 5000 cP. In terms of brightness, the higher value of the white field, the higher level of material brightness.

2.2 Method

2.2.1 Edible Film Making Process

Alginate-based edible film was processed according to the method applied by Murdinah (2007), through modification of the types and concentration of main ingredients namely alginate and starch as well as the use of sorbitol as plasticizer. The three types of starch were canna starch, mocaf starch and breadfruit starch, meanwhile the starch concentrations levels were 0%, 0.1%, 0.3% and 0.5%. Alginate and starch according to the type and concentration level of use were mixed with distilled water in a beaker glass and stirred. The mixture was then heated to 70ºC. Sorbitol was poured according to the formulation into the solution while still stirring the beeswax was subsequently added and heated at 70ºC. The film solution was then transferred into 16 x 16 cm acrylic plates. The plates were allowed to cool at room temperature for 24 hours and dried in an oven at 45ºC for 24 hours. The plates were taken out from the oven and the films were then peeled. The films known as alginate-based edible films were put in a plastic clip and stored in a desiccator at room temperature.

2.2.2 Experimental Design and Data Analysis

This research employed Completely Randomized Factorial Design (CRFD) with two factors, i.e. types of starch (A1 = Canna, A2 = Mocaf, A3 = Breadfruit) and concentration levels of starch (B1 = 0 %, B2 = 0,1%, and B3 = 0,3%, and B4 = 0,5%). All experiments were run with three replications. The data were analysed using Univariate Analysis of Variance (ANOVA) with post host test Tukey's, and the level of significance was set at α<0.05.

2.2.3 Edible Film Characterization

Moisture Content. The moisture content of alginate-based edible films was determined following the method described by (Farhan & Hani, 2017). Edible film samples (2 cm × 2 cm) were cut each of the three random positions and then placed on a porcelain dish. The edible films were dried in an oven at 103-105°C until a constant weight obtained. The moisture content of edible films was calculated from the weight lost after drying and initial weight of edible films.

Thickness. A digital micrometer (Mitutoyo) with 0,001 mm accuracy level was used to measure the thickness of alginate-based edible films. Thickness of ten different random spots of each films were measured, and the averages values in millimeters (mm) were reported as thickness.

Tensile Strength and Elongation. Tensile strength and elongation of alginate-based edible films were measured using TAXT Plus - Texture Analyzer (Balqis, et al, 2017), with slight modification. Edible films were cut into 2 cm x 15 cm. The measurements were conducted under following condition: 10 cm initial distance of grip separation, 3 mm/s test speed, and 3 g trigger forces. Tensile strength (force/initial cross-sectional area) and elongation at break were determined using Texture Expert V.1.15 software package.

Water Solubility. Water solubility of alginate-based edible film was measured using a method described by Murni, et al. (2013). Edible film samples (~3x3 cm^2) were weighed, placed in aluminium cups contained 50 ml distilled water, and then the cups contained films were heated in oven at 100°C for 30 minutes. In order to avoid evaporation of water, the cups were tightly closed and kept at 25°C for 24 h and occasionally shaken. Undissolved films matters were dried in an oven at 40°C until the constant weight achieved. The water solubility of the film was determined according to the ratio of weight of diluted film matter with initial films weight.

Water Vapor Transmission Rate. Water Vapor Transmission Rate (WVTR) of alginate-based edible films were determined according to the slightly modified methods as described by Razzag, et all (2016). WVTR was gravimetrically measured using metal cups equipped with an exposed area metal disc. Edible films were sealed on metal cups containing distilled water, and then metal cups were placed in an incubator (37°C, ±24 %RH) for 24 h. The water loss was measured. The WVTR is the mass of water loss rate at the specific area for 24 h ($g/m^2/24h$).

Morphological Test. Morphological analysis of the cross section of the alginate-based edible films was carried out using SEM (Scanning Electron Microscopy) - JEOL JSM-6360LA referring to a method as described by Setiani, et al (2013) and modified in term of the size of the zoom. Edible film samples were affixed to the set holder which had double adhesive, then gold metal is coated under vacuum. The sample was subsequently put into the SEM, then the topographic image was observed and magnified 400-500 times.

3 RESULTS

3.1 Characteristics of Edible Film

3.1.1 Moisture Content

Based on Table 2, it is recognised that the higher the concentration of the starch used, the higher moisture content of alginate-based edible films will be. It was probably due to the nature of the hydrophilic starch molecule, thus the higher the starch concentration added the higher the moisture content of the alginate-based edible film. Results of Tukey test showed that the moisture contents of alginate-based edible film prepared with various starch concentrations were significantly different each other at $\alpha<0.05$.

Table 2: Moisture content of alginate-based edible film (%).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	8.37± 0.32	9.33± 0.02	9.34± 0.24	9.65± 1.95	9.17± 0.56
Mocaf	8.37± 0.32	8.97± 0.31	9.18± 0.66	9.19± 0.15	8.93± 0.39
Breadfruit	8.37± 0.32	9.01± 0.12	9.22± 0.25	9.27± 0.43	8.97± 0.41
Average	8.37± 0.00^{a}	9.10± 0.20^{ab}	9.25± 0.08^{b}	9.37± 0.25^{b}	

Murdinah, et al. (2007) informed that moisture contents of edible films made from alginate, gluten and beeswax composites were ranging from 21.95 to 24.63%. Edible film prepared from *Aloe vera* with breadfruit flour and canna had moisture content ranging from 4.22 to 22.20% (Afriyah et al., 2015). Compared to those moisture contents, alginate-based edible films from this study had lower moisture contents, i.e. in the range of 8.37 - 9.65%.

3.2 Thickness

Statistically, types of starch (canna, mocaf, breadfruit) significantly affected the thickness of the alginate-based edible film. There are two types of starch polymers namely amylose and amylopectin. The difference in the structure of the two polymers influenced the functional properties of starch (Teti, 2006). Increasing edible film thickness was also affected by the unique colloidal compound properties as thickener and suspender, and the interaction between edible film constituent components (Galus and Lenart, 2013).

Likewise, the higher the starch concentration used, the higher the thickness of alginate-based edible films. It was probably due to the higher the amount of ingredient used in the film matrix, the higher the total solids. McHugh, et al (1994) noted that film thickness was mainly influenced by the concentration of dissolved solids in the film-forming solution.

Table 3: Thickness of alginate-based edible film (mm).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	0.06± 0.00^{a}	0.06± 0.00^{a}	0.06± 0.01^{a}	0.06± 0.00^{ab}	0.06± 0.00^{a}
Mocaf	0.06± 0.00^{a}	0.06± 0.00^{a}	0.06± 0.00^{ab}	0.07± 0.01^{ab}	0.06± 0.01^{a}
Breadfruit	0.06± 0.00^{a}	0.08± 0.01^{c}	0.10± 0.01^{cd}	0.10± 0.01^{d}	0.08± 0.02^{b}
Average	0.06± 0.00^{a}	0.07± 0.01^{b}	0.07± 0.02^{ab}	0.08± 0.02^{c}	

Tukey test results showed that the thickness of alginate-based edible film processed using canna, mocaf and breadfruit starches with different concentration levels were significantly different each other at α <0.05. There were significant interaction effects between starch type and starch concentration on the thickness of alginate-based edible film.

As shown in Table 3, the thickness of alginate-based edible films was in the range of 0.0557 - 0.1028 mm. Those values were comparable to the thickness of edible film produced by Murdinah, et al (2007) using alginate, gluten and beeswax composites as raw materials, i.e. 07 - 0.11 mm. According to Japanese Industrial Standard, the maximum film thickness value is 0.25 mm. So, the alginate-based edible film produced in this study is in accordance with the quality standards set by the Japanesse Industrial Standard.

3.3 Tensile Strength

Edible films with high tensile strength are required for packaging food products to provide protection of food during handling, transportation and marketing (Pitak and Rakshit, 2011). Table 4 showed that breadfruit starch produced alginate-based edible film with the lowest tensile strength compared to others. It was also revealed that the higher the concentration of starch used, the higher the tensile strength of edible film. The higher use of starch provided a stronger structure to the film matrix so that the tensile strength value was high. It was possible that polysaccharides could function in maintaining the cohesiveness and stability of edible films. Increased tensile strength due to increased starch concentration probably related to the presence of amylose and amylopectin, in which both components played an important role in the formation of edible films. Amylose levels increased with increasing starch concentration.

The results of Tukey test showed that the tensile strength of alginate-based edible films processed using canna, mocaf, and breadfruit starches with various starch concentration levels were significantly different each other at α <0.05. There were significant interaction effects between starch type and starch concentration on the tensile strength of edible films.

Table 4: Tensile Strength of alginate-based edible film (MPa).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	1.90± 0.05^{a}	2.75± 0.56^{a}	4.63± 0.99^{b}	4.95± 0.21^{b}	3.56± 1.47^{b}
Mocaf	1.90± 0.05^{a}	4.15± 0.27^{b}	4.69± 0.53^{b}	5.21± 0.79^{b}	3.99± 1.46^{b}
Breadfruit	1.90± 0.05^{a}	2.10± 0.19^{a}	2.32± 0.19^{a}	2.78± 0.38^{a}	2.28± 0.38^{a}
Average	1.90± 0.00^{a}	3.00± 1.05^{b}	3.88± 1.35^{c}	4.31± 1.33^{c}	

Table 4 showed that tensile strength values of alginate-based edible film processed using alginate and various starches were in the range of 1.90-5.21 MPa. Those tensile strength values were greatly different compared to the tensile strength values of edible films produced by Murdinah, et al. (2007) and Afriyah et al. (2015). Tensile strength of edible films made from alginate, gluten and beeswax composites was 13.41-34.87 MPa (Murdinah, et al., 2007). While, tensile strength of edible film using aloe vera, breadfruit flour and canna flour as raw materials was in the range of 1.76-16.23 MPa (Afriyah, et al., 2015). According to the Japanese Industrial Standard, the minimum tensile strength value of edible film is 3.92 MPa. In regard to that standard, alginate-based edible film produced using breadfruit starch in this study is not in accordance with the standard, on the other hand the ones prepared using mocaf and canna starches meet the Japanese Industrial Standards.

3.4 Elongation

Elongation is the percentage increase in the length of the alginate-based edible film when pulled until torn or broken. Table 5 shows that the higher the concentration of starch used, the higher the elongation of alginate-based edible film. Increasing the concentration of starch in edible film formulation resulted in increased elongation value. High amylose content and the addition of plasticizer was suspected to cause a high elongation of edible film. This indication is supported by the fact that a flexible and strong film can be made from the starch containing amylose, while amylopectin provides stability and elasticity in the formation of edible films. Elongation of alginate-based edible film using 0.5% breadfruit and 0.5% canna starch was significantly different from the others at $\alpha < 0.05$.

Table 5: Elongation of alginate-based edible film (%).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	4.60 ± 0.10^{a}	21.13 ± 0.80^{c}	29.23 ± 0.59^{d}	33.00 ± 2.74^{e}	21.99 ± 12.61^{c}
Mocaf	4.60 ± 0.10^{a}	20.67 ± 0.85^{c}	28.53 ± 1.97^{d}	28.90 ± 1.57^{d}	20.68 ± 11.37^{b}
Breadfruit	4.60 ± 0.10^{a}	7.23 ± 0.40^{a}	7.80 ± 0.26^{a}	15.73 ± 0.87^{b}	8.84 ± 4.80^{a}
Average	4.60 ± 0.10^{a}	16.34 ± 0.79^{b}	21.85 ± 0.94^{c}	25.88 ± 1.73^{d}	

Murdinah et al. (2007) reported that elongation of edible films made from alginate, gluten and beeswax composites was ranging from 1 - 2.5%. Afriyah, et al. (2015) noted that elongation of edible films made of aloe vera, breadfruit flour and canna was in the range of 9.23-50.22%. Meanwhile, elongation of alginate-based edible film produced in this study was comparable to both above results, i.e. in the range of 4.60 - 33.0%. Japanese Industrial Standard mentioned that the minimum film elongation value of edible film is 5%. Alginate-based edible film produced in this study is in accordance with the Japanese Industrial Standard.

3.5 Water Solubility

Table 6 shows that the water solubility value of alginate-based edible films produced using various starch concentrations was insignificantly difference, but the trend informed that the higher the concentration of starch used, the lower the water solubility of alginate-based edible film. Edible films with high water solubility values were suitable to be used in ready-to-eat food products because they dissolve easily when consumed (Pitak and Rakshit, 2011).

Table 6: Water solubility of alginate-based edible film (%).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	99.20 ± 0.23^{e}	97.33 ± 0.31^{bc}	97.20 ± 0.10^{b}	95.07 ± 0.32^{a}	97.20 ± 1.69^{a}
Mocaf	99.20 ± 0.23^{e}	98.83 ± 0.93^{cde}	98.23 ± 0.51^{bcde}	97.93 ± 1.07^{bcde}	98.55 ± 0.57^{b}
Breadfruit	99.20 ± 0.23^{e}	99.10 ± 0.30^{de}	97.83 ± 0.47^{bcde}	97.63 ± 0.46^{bcd}	98.44 ± 0.82^{b}
Average	99.20 ± 0.20^{c}	98.42 ± 0.95^{b}	97.75 ± 0.52^{b}	96.88 ± 1.57^{a}	

Statistically, the type of starch, concentration of starch and their interactions significantly affected the water solubility of alginate-based edible films. Siswanti, et al (2009) reported that the water solubility of edible film processed from iles-iles and maizena glucosamanan composite was 50.58%. Alginate-based edible film produced in this study had a higher water solubility compared to that value, i.e. 95.07 - 99.20%. Low water solubility value is one of the important requirements for edible film, especially for use as food packaging of high moisture content and high water activity products (Singh, et al, 2014).

3.6 Water Vapor Transmission Rate (WVTR)

WVTR is the amount of water vapor that passes through the film surface per area (Fransiska, et al, 2018). Table 7 shows that the WVTR of alginate-based edible film produced from breadfruit starch was significantly different from alginate-based edible films processed using canna and mocaf starches. The significant effect was shown by the fact that the higher concentration of starch used, the lower the WVTR of alginate-based edible films.

Results of Tukey test revealed that WVTR of alginate-based edible films using starch concentrations of 0.1%, and 0.3% was insignificantly different, but WVTR of alginate-based edible films using starch concentration of 0% and 0.5% was significantly different. WVTR of alginate-based edible films in this study showed a trend to decrease with increasing starch concentration levels. The decrease of WVTR was probably due to due to a stronger polymer bonding with the increase of the starch concentrations used. Increasing the bond strength between polymers will reduce the water vapor transmission of edible films to gas, vapor and porosity, so that the function of edible film as a barrier to water vapor entry will increase (Pramadita, 2011).

Table 7: Water vapor transmission rate of alginate-based edible film (g/m^2/24 hours).

Type of Starch	Concentrations (%)				Average
	0	0.1	0.3	0.5	
Canna	1513.94± 339.05	2191.58 ±204.70	2374.17± 471.42	1997.23± 162.50	2019.23 ±370.35^a
Mocaf	1513.94± 339.05	2326.72 ±284.84	2374.95± 156.83	1709.29± 366.58	1981.22 ±434.62^a
Bread fruit	1513.94± 339.05	2698.67 ±235.06	3048.75± 192.41	2047.43± 118.62	2327.20 ±682.71^b
Average	1513.94± 0.00^a	2405.66 ±262.60^c	2599.29± 389.24^c	1917.98± 182.47^b	

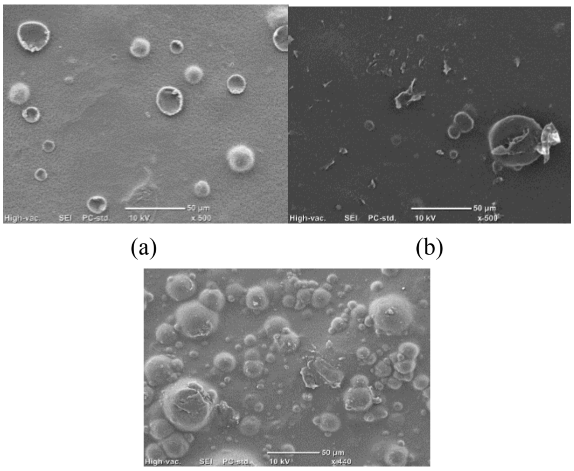
Murdinah, et al (2007) noted that WVTR of edible films made from alginate, gluten and beeswax composites was ranging from 154.34 - 284.40 g/m^2/ 24 hours. Afriyah, et al (2015) obtained that WVTR of edible film formulated using aloe vera, canna flour and breadfruit was in the range 2.83 - 4.66 g/m^2/24 hours. WVTR of alginate-based edible film developed in this study was ranging between 1513.94 – 3048.75 g/m^2/24 hours, and those values were pronouncedly higher compared the above studies.

3.7 Morphological Test

Scanning Electron Microscope (SEM) is a tool that can form surface shadows microscopically. SEM technique is a surface examination and analysis (Wirjosentono, 1995). Results of surface morphology analysis of alginate-based edible film can be seen in Figure 1.

The results of SEM test on alginate-based edible film with the addition of canna starch (Fig 1.a) revealed that the molecular structure of the alginate-based edible film surface was less smooth, in which starch granules and beeswax were still found. A rather rough surface of edible was due to beeswax which was insoluble in water. The result of SEM test on the alginate-based edible film with the addition of mocaf starch showed that the surface structure of the alginate-based edible film molecule looked quite smooth and flat but a few starch granules was still encountered (Fig. 1.b).

Whereas the result of SEM test on alginate-based edible film with the addition of breadfruit starch (Fig. 1.c), indicated that the surface of the film was rough and many starch granules mixed with other components such as water and sorbitol were still found. The size of breadfruit starch granules was larger than the other starches because the mixture in the alginate-based edible film was difficult to dissolve.



(a) (b) (c)

Figure 1: Morphological test on the surface of alginate-based edible film and (a) Canna starch; (b) Mocaf starch, and (c) Breadfruit starch.

4 CONCLUSIONS

The type of starch used for manufacturing alginate-based edible film, namely canna, mocaf, and breadfruit starches significantly affected the mechanical and physical properties of the edible films at α<0.05. The type of starch significantly affected thickness, tensile strength, elongation, water solubility and WVTR of alginate-based edible films. The recommended type of starch for producing alginate-based edible film was mocaf starch.

The starch concentrations significantly influenced the characteristics of alginate-based -at α<0.05. Moisture content, thickness, tensile strength, elongation, water solubility and WVTR of alginate-based edible films were pronouncedly influenced by starch concentration levels. Alginate-based edible films were suggested to use 0.5% starch to obtain the best quality.

Interactions between starch types and starch concentrations significantly affected on the quality of alginate-based edible films at significant level <α (0.05). Thickness, tensile strength, water solubility and elongation of alginate-based edible films were markedly affected by the interaction of starch type and starch concentration. The best alginate-based edible films was obtained by using 0.5% mocaf in their formulation. Characteristics of the film were 9.19% moisture content, 0.0713 mm thickness, 5.21 MPa tensile strength, 28.90% elongation, 97.93% water solubility and 1709.287 g / m2 / 24 hours WVTR. The surface properties of the film according to SEM test were quite smooth and dense, although few starch granules were still found.

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