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Formulation and Characterization of Edible Film with Concentration Various of Sodium Carboxymethyl Cellulose from Kepok Banana (*Musa paradisiaca* L.) Bark and Plasticizer

Surya Ningsi*, Nurfahima Abbas, Muhammad Ikhlas Arsul

Universitas Islam Negeri Alauddin, Makassar, Sulawesi Selatan, Indonesia

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*Corresponding author e-mail: inci_far@yahoo.com

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ABSTRACT

Introduction: *Bark of kepok banana (Musa paradisiaca L.) contains high cellulose that can be synthesized into sodium carboxymethyl cellulose (Na-CMC). Na-CMC can be used as a raw material for the preparation of edible film.* ***Aims:*** *The purpose of this study was to determine the concentration of sodium carboxymethyl cellulose from kepok banana bark combine with plasticizer to produce edible films with good characteristics and quality.* ***Methodology****: Edible films were made with varying concentrations of 6%, 10%, and 14% Na-CMC; 4% glycerol and 9% sorbitol as plasticizer. Edible films were made using casting method and characterized including organoleptic test, thickness, pH, water absorption, elongation, and tensile strength.* ***Result****: Edible film made is in the form of thin layer, transparent white in color, and odorless; thickness 0,10±0,01-0,38±0,01 mm; pH 7,1±0,08-7,7±0,04; moisture content 35,47-91,97%; elongation 16,33±3,66-53,29±6,13%; and tensile strength 0,0528±0,00-3,9871±0,04 MPa..* ***Conclusion:*** *Different concentrations of Na-CMC and plasticizer affect the characteristics produced. The best result based on Japanese Industrial Standard (JIS) is formula F5 with 10% Na-CMC concentration and 9% sorbitol as plasticizer.*

KEYWORDS: *Kepok banana, edible film, Na-CMC, plasticizer, glycerol, sorbitol*

INTRODUCTION

Banana kepok bark containt 64% of cellulose (Noviratri, 2018) . The high content of cellulose can makes it a very potential material to be used as a renewable material, for example through processing into sodium carboxymethyl cellulose (Na-CMC) (Prasetya, Istiqomah, & Yamtana, 2017). Na-CMC is a derivative of cellulose and is widely used in the world of food industry, pharmaceuticals, and cosmetic products. In addition, Na-CMC also functions as a thickener, stabilizer, binder, and as a raw material for making edible films (Angor, 2016).

The advantages of Na-CMC as a raw material in edible film production include its economical aspects which are more favorable compared to other materials such as lipids or proteins, odorless, consumable, abundant availability, its ability to dissolve in water which facilitates the process of making edible films, and has a high molecular weight to produce films with sufficient mechanical strength to withstand the load (Tongdeesoontorn, Mauer, Wongruong, Sriburi, & Rachtanapun, 2011).

Edible film, as a packaging technology that is consumable, safe, and environmentally friendly, is a thin layer with a thickness of less than 0.25 mm. The main function of edible film is as a mass transfer barrier, especially in controlling moisture transfer that can affect the quality and safety of packaged products (Warkoyo, Taufani, & Anggriani, 2021). In the pharmaceutical, edible film has the potential to be used as a material for making capsule shells (Darni & Rakhman, Aplikasi Edible Film Dari Rumput Laut Eucheumma cottoni dan Pati Sorgum dengan Plasticizer Gliserol dan Filler CaCO3sebagai Bahan Pembuat Cangkang Kapsul, 2017).

There are two main components for edible film formulation, it is polymers and solvents (Garcia, 2017). However, when making edible films using a single material, the resulting films tend to have brittle and stiff properties. To overcome this problem, the addition of plasticizers is required. Plasticizers are chemical compounds that are added into polymeric materials to change the physical properties of the material, such as increasing elasticity and reducing stiffness in edible films. Glycerol and sorbitol are often used as plasticizers to function optimally due to their ability to break internal hydrogen bonds in intramolecular bonds in the film, thereby increasing flexibility (Mujiarto, 2005).

Glycerol has been recognized by the Food and Drug Administration since 1959 as a safe ingredient for use in various food applications. Glycerol is hydrophilic, which means it is able to attract water molecules into the edible film solution matrix, thus increasing the thickness and elasticity of the film (Garcia, 2017). Meanwhile, the use of sorbitol as a plasticizer is also effective in making edible films due to its ability to reduce the diffusion rate of gases such as oxygen, thereby, reducing the entry of oxygen into the wrapped foodstuff (Hidayati, Zuidar, & Ardiani, Aplikasi Sorbitol Pada Produksi Biodegradable Film dari Nata De Cassava, 2015).

Based on this description, edible films with good quality characteristics can be made using variations in Na-CMC concentration. The purpose of this study was to formulate and characterize edible films with variations in the combination of Na-CMC with glycerol and sorbitol as plasticizer.

MATERIAL AND METHODS

Material

Na-CMC made by banana kepok bark obtained from UIN Alauddin, glycerol (Pharmaceutical grade, Brataco), sorbitol (Pharmaceutical grade, Brataco), sodium hydroxide 10% (b/v) (Pharmaceutical grade, Brataco), acetic acid 10% (v/v) (Pharmaceutical grade, Brataco), sodium chloride (NaCl) (Pharmaceutical grade, Brataco), ethanol 96% (Pharmaceutical grade, Brataco), sodium hypochlorite (Pharmaceutical grade, Brataco).

Table 1. Edible film formulas

Material	Formula (%)						Function
	F1	F2	F3	F4	F5	F6	
Na-CMC	6	10	14	6	10	14	Membrane
Glycerol	4	4	4	-	-	-	*Plasticizer*
Sorbitol	-	-	-	9	9	9	*Plasticizer*
Aquadest	Add 100 ml						Solvent

Preparation Of Film

respective formulas (Table 1). The mixture was stirred for 30 minutes where the temperature was maintained at 70℃. The mixture was then poured into molds and then dried at 50^{o}C (Hidayati, Satyajaya, & Zulferiyenni, 2019).

Characterization

Organoleptik

Organoleptic observations include shape, color, and odor of the edible film (Tyas, Meinitasari, Safitri, Septiningrum, & Nila, 2018).

Thickness

Film thickness was measured using a micrometer. Average thickness values were obtained from measurements taken at five different locations (i.e. each at the edge of a rectangle and in the middle) (Setiani, Sudiarti, & Rahmidar, 2013).

pH

Measurement of edible film pH is done using a pH meter by dissolving 1 g of edible film with 10 mL aquadest (Tyas, Meinitasari, Safitri, Septiningrum, & Nila, 2018).

Moisture content

The moisture content (MC) was measured using the procedure outlined by Fera and Nurkholik (Fera & Nurkholik, 2018) with slight modification. Samples of the film measuring 2 cm×2 cm were placed in pre-dried weighing bottles in triplicate and then subjected to 24 hours of oven drying at 90 °C in an electric oven with forced convection (DHG-9140A, Yiheng Scientific Instrument Co. Ltd., Shanghai, China). The value of moisture content was calculated using the following equation:

$$MC\ (\%) = \frac{W - W_0}{W_0} \times 100\%$$

where W and W_0 are the initial and final weights of film samples, respectively.

Elongasi

The elongasi test using the IK-MT-30.70 test method, percent elongation at break was calculated on the basis of length extended as compared to the original length of the film (Fera & Nurkholik, 2018).

Tensile strength

The films' mechanical properties, including tensile strength (TS), were assessed following the standard ASTM method D 882-88 using an Instron Universal Testing Machine (Model

5565, Instron Engineering Corporation, Canton, MA, USA) equipped with a 0.5 kN load cell. Precision double blade cutter (model LB.02/A, Metrotec, S.A., San Sebastian, Spain) was used to cut rectangular strips (2.54 × 10 cm) from individually prepared film. The test setup involved setting the initial grip separation at 50 mm and cross-head speed at 100 mm/min. Tensile strength (t) was calculated by dividing the maximum load (Fmax) by the initial cross-sectional area (A) of the film sample (Setiani, Sudiarti, & Rahmidar, 2013).

Tensile strength can be calculated using the following equation:

$$t = \frac{Fmax}{A}$$

RESULTS AND DISCUSSION

The kepok banana consists of several parts, namely roots, stem, leaves, and fruit. Of all the parts of the banana plant, the part that is rarely utilized by the community is the banana stem. The utilization of this part of the banana plant is still very limited (Agustina, 2008). One of the contents that can be utilized in banana stump is cellulose. Ningsi et al., using kepok banana stem, has a high cellulose content of 64.9% so that it can be used as an excipient in pharmaceutical preparations (Ningsi, Tahar, Syahrana, & Arifin, 2020). Cellulose has several derivatives that are widely used in the pharmaceutical industry, one of which is sodium carboxymethyl cellulose (Na-CMC) which can be used as raw material in the manufacture of edible films (Angor, 2016). In

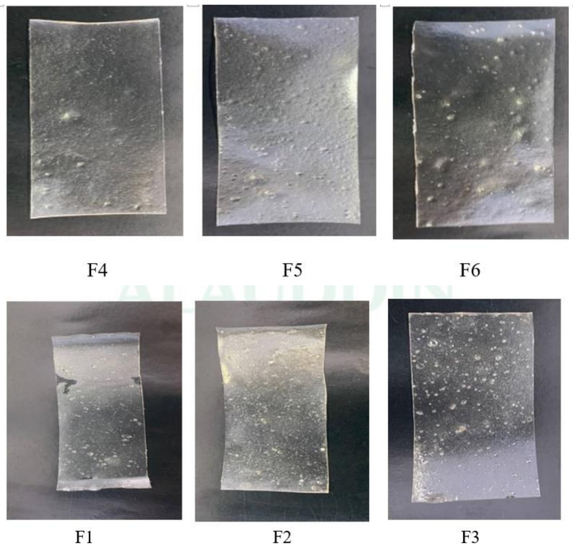

Figure 1. Edible film formulas

this study, Na-CMC used was derived from the formulation of Na-CMC derived from banana kepok cellulose.

One of the uses of edible film is that it can be used as raw material for making capsule shells (Darni & Rakhman, 2017). This study uses three edible film constituents, namely Na-CMC, plasticizers (glycerol and sorbitol), and solvents. The manufacture of edible films with the use of a single material still leaves several shortcomings including brittle and rigid properties, this can be overcome by the addition of plasticizers (Hidayati, Zuidar, & Ardiani, 2015).

Organoleptic test is a test conducted by observing the shape, color, and smell of the edible film produced (Tyas, Meinitasari, Safitri, Septiningrum, & Nila, 2018). Edible film dries for 48 hours and can be seen in Figure 1. Organoleptic test results from edible films F1, F2, F3, F4, F5 and F6 obtained results in the form of a thin layer, transparent white color and odorless.

Table 2. Characterization of edible film

Formula	Thickness (mm)	pH	Moisture content (%)	Elongation (%)	Tensile strength (MPa)
FI	0,10 ± 0,01	7,2 ± 0,04	91,97	16,33 ± 3,66	0,0528 ± 0.00
F2	0,12 ± 0	7,5 ± 0,08	61,56	35,43 ± 7,57	0,3090 ± 0.00
F3	0,18 ± 0,02	7,7 ± 0,04	39,13	37,66 ± 5,03	0,1836 ± 0, 06
F4	0,12 ± 0	7,1 ± 0,08	73,15	29,32 ± 2,21	0,2876 ± 0,04
F5	0,14 ± 0,01	7,3 ± 0,04	38,82	53,29 ± 6,13	3,3681 ± 0,02
F6	0,38 ± 0,01	7,6 ± 0,13	35,47	37,25 ± 2,94	3,9871 ± 0,04

n = 3

The thickness test aims to determine the ability of edible film to prevent the exchange of substances either from the environment or the product that will be packaged later. The thickness test can be seen in Table 2 where formulas F1, F2, F3, F4, and F5 meet the requirements while F6 does not meet the requirements. The edible film thickness test requirement based on Jappanese Industrial Standard (JIS) is a maximum of 0.25 mm (JIS, 1975). Edible films that have a thickness of > 0.25 mm have poor physical properties because they can cause the edible film produced to be brittle and stiff. In this study, the thickness of edible film increased with increasing concentration of Na-CMC and plasticizer used. (Nurindra, 2015) reported that increasing the concentration of total soluble solids in the edible film suspense will result in the structure of the polymers that make up the film also increasing so that the thickness of the film will increase.

The same thing was also found in research conducted by Sanyang (2016) that the type of plasticizer used also affects the thickness of edible film because of the constant concentration of Na-CMC in the formulation and also the use of glycerol as a plasticizer has a lower level of polarity compared to sorbitol, therefore the use of sorbitol plasticizer produces thicker edible film compared to edible film with glycerol plasticizer (Sanyang, Sapuan, Jawaid, Ishak, & Sahari, 2016). In accordance with research conducted by Ghasemlou, (2011) that films with sorbitol plasticizers produce edible films that are thicker than glycerol plasticizers because sorbitol added to edible films will cause water bound in edible films to be greater (Ghasemlou, Khodaiyan, & Oromiehie, 2011). According to Raharjo (2012) sorbitol has hydrophilic properties, can bind water so as to increase the thickness of the edible film (Raharjo, Dewi, & Haryani, 2012).

In this study, the thickness of the edible film also affects water absorption, namely the thicker the edible film, the weaker the barrier properties or absorption ability because the structure is tighter. In line with research conducted by Hasdar et al., that the thicker the edible film, the more it will affect mass transfer and water absorption (Hasdar, Erwanto, & Triatmojo, 2011). This also shows that the edible film produced can be used as a capsule shell, in accordance with research conducted by Aulia & Wafiroh that edible films with low water absorption can be used to make capsule shells to be applied to the digestive system in the intestine (Hikmah & Wafiroh, 2023).

The pH test aims to see the acidity of the preparation. The pH of edible film refers to the pH of the intestine, which ranges from 7-8 (Hikmah & Wafiroh, 2023). In this study, of the six formulas, the pH obtained ranged from 7.2-7.7. The pH test results can be seen in Table 2, so it can be concluded that the edible film produced has the potential to be used as a capsule shell to be applied in the intestine. The difference in Na-CMC concentration does not really affect the pH value.

Water absorption or water resistance test is one of the tests that aims to determine the level of edible film resistance to water absorption. This test is conducted to see the ability of edible film to protect the product from water. The results of the absorption test can be seen in Table 2 where formulation F6 has the lowest water absorption of 35.47% and formulation F1 has the highest water absorption of 91.97%. In formula F1 causes the edible film to break easily and decompose during immersion and in formula F6 has the lowest water absorption, this is because formula F6 is the thickest formula. The thickness of the edible film affects the water absorption, namely the thicker the edible film, the barrier properties or absorption ability are weaker because the structure is tighter. In line with research conducted by Hasdar et al., that the thicker the edible film, the more it will affect mass transfer and water absorption. (Hasdar, Erwanto, & Triatmojo, 2011).

The difference in the type of plasticizer added to the edible film will affect the water absorption. This is in line with research conducted by Zahra et al., which states that the type of plasticizer on the surface of the edible film will control the absorption of liquids or solids so that it affects the water absorption of the edible film. In addition, another factor causing the high value of water absorption in edible film is its constituent components. The content of Na-CMC in edible film affects water absorption as the Na-CMC content increases due to an increase in the number of hydroxyl groups (Zahra, Finadzir, & Yulistiani, 2020). Where Na-CMC is able to reduce the diffusion of water molecules in edible film (Yusriah, Sapuan, Zainudin, & Mariatti, 2012). This is in accordance with research conducted by Rusli et al., that the higher the concentration of Na-CMC, the lower the water absorption or in other words, the decrease in water absorption in edible films tends to be inversely proportional to the increase in Na-CMC concentration due to soluble solids derived from Na-CMC and increasing the number of molecules in the edible film solution (Rusli, Metusalach, Salengke, & Tahir, 2017).

Percent elongation is the maximum change in length at the time of stretching until the film sample is cut off. The elongation test results can be seen in Table 2 where there is an increase in elongation as the Na-CMC concentration increases. Na-CMC has a high gel strength content so that the use of large amounts will cause better water binding ability, this provides a gel matrix to increase the elongation of the edible film. However, the same thing does not apply to formula F6, which is a decrease in elongation. The decrease in the elongation percent in formula F6 is caused by factors such as less homogeneous mixing so that the insertion of plasticizer material into the film matrix has not taken place perfectly to reduce the elongation percent of the edible film (Wardah & Hastuti, 2015). As the tensile strength value increases, the flexibility of the film decreases so that it decreases the percentage of elongation, this also occurs because the drying time is too long, and the pressure is sometimes inconsistent (Wirawan, Prasetya, & Ernie, 2012).

The tensile strength test aims to determine the size of the specific edible film strength or the maximum pull that can be achieved until the film remains before breaking / tearing. The greater the tensile strength, the better the edible film is in resisting mechanical damage (Nairfana & Ramdhani, 2021). The results of the tensile strength test can be seen in Table 2, none of the six formulas meet the requirements of SNI 7818: 2014, which ranges from 24.7 Mpa-302 MPa. However, formulas F5 and F6 meet the requirements based on JIS (Japanesse Industrial Standart), which is at least 0.392 MPa (JIS, 1975) The tensile strength in this study increased along with the increase in Na-CMC concentration. In formula F2, there was an increase in tensile strength of 0.309 MPa, but then a decrease in tensile strength in formula F3, which was 0.1836 MPa. This is the maximum film formation condition to achieve the highest tensile strength value. The maximum condition shows that Na-CMC reacts properly with the plasticizer so that it can increase the tensile strength value. The higher the concentration of Na-CMC, the tensile strength decreases because the molecular structure of edible film is amorphous, namely branched chains that are not tightly arranged so that the distance between molecules is greater and the strength of molecular bonds becomes weaker as a result of the lower the force needed to break the edible film. If the concentration is higher, Na-CMC is unable to bind the plasticizer, so the resulting film is hard and brittle. This condition will reduce the intermolecular forces between polymer chains in CMC and the main raw materials used. This is in line with research conducted by Putri & Desi which states that the concentration of Na-CMC affects the tensile strength of edible films, namely the higher the concentration of Na-CMC used, the tensile strength value will decrease after reaching the highest tensile strength limit (Putri & Desi, 2019).

The addition of plasticizers can also reduce the internal hydrogen bonds of molecules and cause a weakening of the intermolecular tensile forces of adjacent polymer chains thereby reducing the breaking strain. These results are also supported in research Sitompul & Zubaidah which states that the addition of plasticizers will cause a decrease in the tensile force between polymers when water evaporation occurs which results in decreased resistance to mechanical treatment of the film (Sitompul & Zubaidah, 2017). Edible films that have high tensile strength will protect the packaged product from mechanical disturbances well (Fardhyanti & Julianur, 2015).

The best result of the six formulas that meet JIS characterization standards is formula F5, which is with 10% Na-CMC concentration using sorbitol plasticizer. Formula F5 is an edible film in the form of a thin layer, transparent white in color, and odorless, having a thickness of 0.14 mm, pH 7.3, water absorption of 38.82%, percent elongation of 53.29%, and tensile strength of 3.3681 MPa.

CONCLUSION

The characteristics of edible film are influenced by variations in Na-CMC concentration and type of plasticizer, where F5 is the best formula that has the potential to be further made into capsule shells.

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