Effect of Sago Starch Concentrations, Stirring Speeds, and Lemongrass Oil Concentration for Edible Film Production Using Solvent Casting Method

H Santosa*, M. Djaeni, Ratnawati, N Rokhati, A P Setiatun, Afriyanti

Chemical Engineering Department, Faculty of Engineering, Diponegoro University, Jl. Prof Sudharto,SH, Tembalang, Semarang, Indonesia
Email*: hersantos@undip.ac.id

Abstract. Foods such as meat, fillet, fruit and vegetable tend to have short shelf life when it is stored outside without packaging. Recently, edible film and coating become a potential alternative for food packaging. Edible film brings a lot of advantages from environmental aspect because it is easier to be decomposed rather than the synthetic film. Furthermore, edible film might be produced from both natural raw material and waste materials. Edible film from natural raw material gives a rigid structure. Another additive agent must be used to make a better quality product, such as glycerol and lemon grass oil as plasticizer and antimicrobial agent, respectively. These materials are commonly used in edible film production due to its capability for extending the food shelf-life by inhibiting bacterial growth. The objectives of this study are to investigate the effect of starch concentration (3% to 10% w/v), stirring speed (350, 700, 1100 rpm) and lemon grass oil concentration (1%, 5% v/v) on tensile strength, water vapor permeability, and antimicrobial activity. Results show that 4% (w/v) sago starch gives the best edible film mechanical properties and least water transfer. Edible film with 4% (w/v) sago starch, 8 rpm constant stirring, and 5% (v/v) lemongrass oil gives the best result in tensile strength, water vapor permeability and bacterial growth. In addition, variables of starch concentration, stirring speed and lemon grass oil concentration are interrelated to produce the best edible film.

Keywords: edible film, lemongrass oil, gliserol, sago starch, solvent casting.

1. Introduction

Starch is commonly used in food industry as emulsifier [1]. Sago starch mainly contains carbohydrate that can be widely utilized in various field, however its applications in industries are not optimal [2]. Edible film is one of developed product which uses carbohydrate from sago starch as raw materials. It is thin barrier made from materials that can be consumed and used to preserve the products. The film also acts as a barrier to mass transfer (e.g moisture, oxygen, and solutes) [3]. Edible film made by starch
resulting a compact structure and low solubility of the film. The advantage of using starch as the main ingredient of edible film is that the resulting a compact structure of film, but it is brittle [4, 5]. There are several methods for edible film production, such as solvent casting, hot melt extrusion, semisolid casting and rolling solvent casting [6]. The Solvent Casting method is most widely used because it is cost-effective. Solvent casting requires a large amount of solvent and uses the principle of gelatinization in the range temperature of 64.5–70 °C [4, 7].

Karim et al. (2011) obtained the optimum sago starch concentration is 4% (w/w) for preparing a low tensile strength and flexibility of the product [8]. The films, which is made from nature raw materials, have a rigid and brittle structures, thus it is necessary to add a plasticizer agents (glycerol) [9]. However, the thickness and moisture content of edible films were influenced by glycerol concentration. The use of 10% glycerol produces edible films produced the best characteristic of films [10].

Given of the previous studies, it is necessary to modify the method thus better properties of edible films would be obtained. The products are expected to have a specific character, anti-microbial properties, so that it has a prolong shelf life. Lemongrass oil is a material which has the capability of it and would be used in this study. The objectives of this study are to investigate the effect of starch concentration (3% to 10% w/v), stirring speed (350, 700, 1100 rpm) and lemon grass oil concentration (1%, 5% v/v) on tensile strength, water vapor permeability, and antimicrobial activity.

2. Method

2.1. Material

Sago starch was purchased from Javara, as one of biodiversity product of PT. Kampung Kearifan Indonesia. Glycerol was produced and purchased by PT. Ecogreen Oleochemicals and CV. Indrasari, respectively. Furthermore, distilled water and lemongrass oil were purchased in CV. Indrasari.

2.2 Preparation of starch / gelatin edible film

Sago starch in varying concentrations (3%, 4%, 5%, 6%, 7%, 8%, 9%, and 10% w/v) was dissolved in 100 ml of distilled water at 700 rpm for 30 minutes. This stage was conducted to determine the best concentration for edible film preparation. Afterwards, 1.5% (v/v) of plasticizer agent (glycerol) was added, heated (up to 70°C), and stirred for 10 minutes. The lemon grass oil (1% and 5% v/v) was also augmented on the solution. The mixture then was casted onto acrylic plates (9 cm x 15 cm x 3 mm) and dried in an oven at 50°C for 5 hours. The dried films were obtained by peeling it off from the casting plate and cooled it in room temperature. The best starch concentration was then mixed with glycerol 1.5% (v/v) and various concentration of lemongrass oil (1% and 5% v/v) at varying stirring speed of 350 rpm, 700 rpm and 1100 rpm.

2.3. Tensile Strength determination

Tensile strength property of edible film was analyzed using ASTM D1894 procedure and texture analyzer device. The samples were placed in a fixture base table, afterwards compressed and hold until it was broken down. The output data was converted to pressure (tension) unit, MPa.

2.4. Water Vapor Permeability (WVP) determination

WVP of the edible film was determined by using ASTM E 96-65 method. The weight of test tubes was recorded and plotted as a function of time. The slope of each line was calculated by linear regression (R² ≥ 0.99). WVP values were calculated using Eq.(1)

\[ WVP = \frac{WTR \times L}{\Delta P} \quad (1) \]
where WVTR is the water vapor transmission rate (g m⁻² h⁻¹) through a film, calculated from the slope of the straight line divided by the exposed film area (m²), while L is the mean of film thickness (mm), and ΔP is the partial water vapor pressure difference (Pa) across the two sides of the film. The measurements were carried out duplicates for each type of film and the final WVP values were averaged.

2.5. Bacterial growth determination
Bacterial growth analyses were conducted using total plate count method in accordance with SNI 01-2332.3-2006. These measurements were observed for 24 hour with temperature of 37°C and the bacteria could be counted.

3. Results and Discussions

3.1. Effect of sago starch concentration on tensile strength and WVP
Tensile strength is the maximum tensile stress sustained by the sample during the tension test and affected by the concentration of sago as the component of edible film solution [11]. The values of tensile strength and WVP of edible films in different sago starch concentration is described in Table 1. Commonly, values tend to rise up along with the increase of starch concentration, although some formulation showed the different pattern. Amylose, which is contained in sago starch, produces hydrogen bond. The higher starch concentration would add number of hydrogen bonds. Furthermore, it could lead edible films becoming more rigid and have a high tensile strength.

Table 1. Tensile strength values of edible film in varying sago starch concentrations

| Sago Starch Concentration [%w/v distilled water] | Tensile Strength [mPa] | WVP (x 10⁻¹²) [g/mPa.s] |
|-----------------------------------------------|------------------------|-------------------------|
| 3                                             | 0.14                   | 5.31                    |
| 4                                             | 5.80                   | 2.92                    |
| 5                                             | 4.80                   | 4.21                    |
| 6                                             | 4.83                   | 8.86                    |
| 7                                             | 17.44                  | 6.65                    |
| 8                                             | 7.61                   | 6.60                    |
| 9                                             | 13.52                  | 0.12                    |
| 10                                            | 7.26                   | 7.90                    |

Furthermore, WVP gives a characteristic of edible film as the ability of film to resist the water vapor that emerge the film. The different concentration between food side and film side would affect the permeability of film. The higher concentration induced a faster mass transfer, thus the product would be shrinking easily and endanger the product quality automatically. Film thickness would probably affect WVP value. Film with high thickness will have a good resistant to the transfer mass. High concentration of sago starch caused more solid formed in film, so it lead the film becoming thicker.

3.2. The best mechanical properties of edible film
Tensile strength and WVP are the main factor which could be used as parameters for choosing the optimum concentration for edible film production [12]. According to Japanese Industrial Standard, a good edible film has the maximum value of WVP (5.5 x10⁻¹³ g/mPa.s) and tensile strength ( > 0.392 N/mm²). Each sample was sorted from smallest to highest to determine the best formulation. Table 2 describes the ranking of tensile strength and WVP. Seven points were given to the nearest value of tensile strength.
standard, while 1 point would be given for the farthest. According to the total scoring, 4% (w/v) induced the best composition for tensile strength and WVP.

Table 2. Ranking of tensile strength and WVP for choosing the best formulation

| Sago Starch Concentration [%w/v distilled water] | Ranking Tensile Strength | Ranking WVP | Total Score |
|-------------------------------------------------|--------------------------|-------------|------------|
| 3                                               | 3                        | 7           | 10         |
| 4                                               | 1                        | 6           | 7          |
| 5                                               | 2                        | 2           | 4          |
| 6                                               | 7                        | 4           | 11         |
| 7                                               | 5                        | 5           | 10         |
| 8                                               | 6                        | 1           | 7          |
| 9                                               | 4                        | 3           | 7          |

3.3. Effect of stirring speed and lemongrass oil concentration on tensile strength and WVP

The mechanical properties of edible film at various lemongrass oil concentrations stirring speed are shown in Table 3. It is found that the higher stirring speed would lead a lower and greater value of tensile strength and WVP. Masamba (2016) reported that homogenization and mechanical properties of edible films would be effected by stirring speeds [13]. A high stirring speed would bring the matrix weak thus causing a decrease value of WVP. Commonly, the value of tensile strength decreased as the increase of lemongrass oil concentration. This is in good agreement with the results of Maizura et al. (2007), who reported that increasing concentration of lemongrass oil in the film also decreased the tensile strength [14]. Lemongrass oil affects the intermolecular interactions among starch molecules by decreasing the film resistance for elongation. Previous research reported that films containing lemongrass oil were less resistant and stretchable in comparison with film without lemongrass oil [15]. Another research also brought the similar result that the increasing concentration of garlic oil in the film also decreased the tensile strength [16].

Lemongrass oil affects water vapor permeability by modification of film structure. However, antimicrobial agents such as lemongrass oil, cinnamon oil, citral, oregano oil did not influence the WVP value of edible film [17]. The effect of lemongrass oil on WVP given that WVP was increase based on increasing of lemongrass oil concentration. The increase of WVP is related to the modification of film structure by the lemongrass oil.

Furthermore, the antimicrobial activity of sago starch film with lemongrass oil was determined by calculating the total colony forming in a unit (Table 3). Film with higher lemongrass oil concentration gave better antimicrobial activity. It was shown that total colony formed when using 5% (v/v) of lemongrass oil was less than the utilization of lemongrass oil 1 (v/v). Chitosan and starch films retained volatile components present in the essential oil less effectively since it only has one type of polymer chain [18]. Therefore, it allowed more volatile components into the vapor phase with resultant decrease in antimicrobial activities of the film. Antimicrobial agent affected tensile strength, water vapor permeability, and so does the plasticizer [19]. Plasticizer also gives a negative effect to antimicrobial activity. Further considerations in antimicrobial packaging choice are the concentration of antimicrobials in polymer film, the effect of film thickness on activity and physical properties and mechanical properties of the polymer. However, if antimicrobial is entrapped into the bulk of the material, thickness plays will play a role in diffusion and concentration at the film’s surface. Incorporating antimicrobial agents may change the film’s mechanical and barrier properties. The additive may change polymer conformation.
altering diffusion or may interact directly with antimicrobial. Lack of bacteria growth under a film may indicate inhibition, but appropriate controls must be included. This may be due to simple restriction to oxygen. The potential reduction in antimicrobial activity due to immobilization must be considered. Antimicrobial may immobilize to other material that cause the decreasing activity in antimicrobial activity. One of important antimicrobial component of the lemongrass is citrus as an acyclic unsaturated monoterpenaldehyde compound [20]. Citrus thought to inhibit microbial growth by damaging the cell membrane. This compound is known as volatile substance which is sensitive with thermal condition. Citrus is found naturally in the volatile oils of citrus fruits, lemongrass, and other herbs and spices [21]. According to the experiment result, antimicrobial activity with 700 rpm stirring speed and 1 (%v/v) had least colony formed in edible film.

| Stirring Speed (rpm) | Lemongrass Oil Concentration [%v/v] | WVP(x 10^{-12}) [g/m.Pa.s] | Tensile Strength [mPa] | Total Plate Count (CFU/ml) |
|----------------------|------------------------------------|-----------------------------|------------------------|---------------------------|
| 350                  | 1                                  | 1.40                        | 12.51                  | 31                        |
| 350                  | 5                                  | 2.63                        | 3.73                   | 110                       |
| 700                  | 1                                  | 1.97                        | 12.90                  | 1                         |
| 700                  | 5                                  | 2.83                        | 3.18                   | 19                        |
| 1100                 | 1                                  | 8.93                        | 0.12                   | 180                       |
| 1100                 | 5                                  | 5.25                        | 2.80                   | 180                       |

4. Conclusions
Sago Starch based edible film characterization needs further knowledge about barrier properties as well as their dependence with sago starch concentration, stirring speed, lemongrass oil concentration and film thickness. In this case, it is shown the concentration of sago starch affects both tensile strength and water vapor permeability in edible film. While stirring speed affects tensile strength and WVP by microstructure of the film. Another aspect that affects water vapor permeability is the film thickness. Tensile strength become higher as the sago starch concentration increased. Antimicrobial agent inhibit growth of bacteria but also affects mechanical and barrier properties of edible film.

Acknowledgement
The authors are grateful for the financial support from Faculty of Engineering Diponegoro University through “Hibah Penelitian Dasar 2018”.

References

[1] Herawati H. 2016, Jurnal Penelitian dan Pengembangan Pertanian.30(1):31-9.
[2] Jading A, Tethool E, Payung P, Gultom S. 2011, Reaktor.13(3):155-64.
[3] Bourtoom T. 2008, International Food Research Journal.15(3):237-48.
[4] McHugh TH, Krochta JM. 1994, J Agric Food Chem.42(4):841-5.
[5] Mali S, Grossmann MVE, García MA, Martino MN, Zaritzky NE. 2005, Food Hydrocoll.19(1):157-64.
[6] Hijriawati M, Febrina E. 2017, Farmaka.14(1):8-16.
[7] Hui YH, Sherkat F. Handbook of Food Science, Technology, and Engineering-4 Volume Set: CRC press; 2005.
[8] Abdorreza MN, Cheng L, Karim A. 2011, Food Hydrocoll.25(1):56-60.
[9] Šuput DZ, Lazić VL, Popović SZ, Hromiš NM. 2015, Food and Feed Research.42(1):11-22.
[10] Arham R, Mulyati M, Metusalach M, Salengke S. 2016, International Food Research Journal.23(4):1669.
[11] Jutaporn C, Suphitchaya C, Thawien W. 2011, International Food Research Journal.18(1).
[12] Nurindra AP. Karakterisasi edible film dari pati propagul mangrove lindur (Bruguiera gymnorrhiza) dengan penambahan carboxymethyl cellulose (CMC) sebagai pemlastis: Universitas Airlangga; 2015.
[13] Masamba K, Li Y, Zhong F. 2016, Food Packaging and Shelf Life.10:97-105.
[14] Maizura M, Fazilah A, Norziah M, Karim A. 2007, Journal of Food Science.72(6):C324-C30.
[15] Péroyal C, Debeaufort F, Despré D, Voilley A. 2002, J Agric Food Chem.50(14):3977-83.
[16] Pranoto Y, Salokhe VM, Rakshit SK. 2005, Food Res Int.38(3):267-72.
[17] Sung S-Y, Sin LT, Tee T-T, Bee S-T, Rahmat A, Rahman W, et al. 2013, Trends Food Sci Technol.33(2):110-23.
[18] Avila-Sosa R, Palou E, Munguía MTJ, Nevárez-Moorillón GV, Cruz ARN, López-Malo A. 2012, International journal of food microbiology.153(1-2):66-72.
[19] Appendini P, Hotchkiss JH. 2002, Innov Food Sci Emerg Technol.3(2):113-26.
[20] Somolinos M, García D, Pagán R, Mackey B. 2008, Applied and Environmental Microbiology.74(24):7570-7.
[21] Supardan MD, Annisa Y, Arpi N, Satriana S, Mustapha WAW. 2016, International Journal on Advanced Science, Engineering and Information Technology.6(2):216-20.