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Effect of Temperature, Concentration, and Type of Plasticizer on Edible Pectin Film Characteristics of Mango Peel (*Mangifera indica L.*)

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ABSTRACT

Plastic is a polymer product that is widely used by the community and is generally non-degradable. Its high usage makes plastic a source of waste that is difficult to decompose. It is important to transform the use of degradable materials as packaging by the community in an effort to overcome plastic waste. The edible film is an environmentally friendly packaging medium, which is generally synthesized from a mixture of pectin, plasticizer, and tapioca starch by heating and stirring. This study aims to study the effect of mixing temperature, pectin concentration, and type of plasticizer on the characteristics of the edible film. The temperature was varied at 70 °C, 80 °C, and 90 °C, with variations in pectin concentrations of 1%, 2%, and 3%. The types of plasticizers used were glycerol, sorbitol, and polyethylene glycol (PEG 400). The characteristics of the edible film were tested from thickness, tensile strength, elongation, and solubility. In the range of variables studied, the optimum conditions for producing the best edible film characteristics were 70 °C, 3% pectin, and the plasticizer of glycerol, giving solubility of 0.670 g water/g edible film, the thickness of 0.392 mm, the tensile strength of 3.200 MPa, and elongation of 0.134.

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1. Introduction

Plastic is a polymer product that is widely used by the community. The high frequency of use has resulted in plastic as a source of waste that causes environmental problems in Indonesia because conventional plastics tend to be non-degradable. In 2019 based on information from the Indonesian Plastic Industry Association (INAPLAS) and the Central Statistics Agency (BPS), plastic waste in Indonesia has reached 64 million tons per year [1]. The transformation of the use of degradable materials as packaging by the community is deemed necessary as an effort to overcome plastic waste. Nowadays, many studies on degradable packaging have been carried out, one of which is edible film. The edible film serves to extend the shelf life of the product because it has antimicrobial and antioxidant functions and is safe for the environment because it can be consumed with the product. Another advantage is that edible film has biodegradable properties that are easy to recycle, and this biodegradable packaging will not produce harmful chemical compounds if burned [2-4].

Edible films are composed of polysaccharides, proteins, and lipids and are equipped with several components that can increase film resistance, such as composites and plasticizers [5]. Commonly used sources of polysaccharides are starch, cellulose, and pectin. Pectin is a type of complex carbohydrate that can be found in fruit and has water-soluble properties. Pectin is useful as a thickener and gelling agent in the manufacture of edible films [6]. Pectin from fruit can generally be found in the skin of the fruit such as durian skin [7], kluwih fruit skin [8], jackfruit skin [9], and mango skin [10].

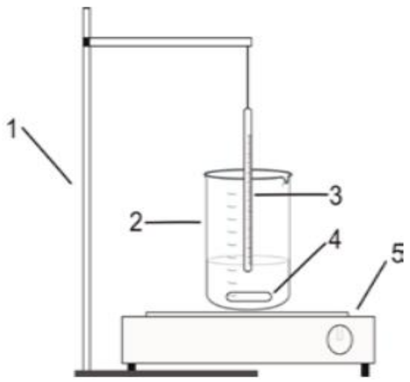
Mango (*Mangifera indica L.*) is a well-known horticultural commodity in Indonesia. According to data from the Central Statistics Agency, the production of mangoes in Indonesia continues to increase every year. It was recorded that the harvest of mangoes in Indonesia from 2016 to 2018 was 1,814,550 tons, 2,203,791 tons, and 2,624,791 tons, respectively. The increase in mango fruit production was followed by an increase in wasted mango skin. Most Indonesian people still consider fruit peels as waste. In fact, mango peel is very potential to be utilized as a source of pectin [11].

In the manufacturing process, edible films also use a plasticizer as additional material to increase the elasticity and extensibility of the film. Plasticizers reduce hydrogen bonds and increase the distance between polymers, resulting in the stretchability or elasticity of the material. To increase flexibility and elasticity, plasticizers with small molecular weight and inert are preferred [4,12]. Plasticizers that are often used are glycerol, sorbitol, and polyethylene glycol. This research studied the manufacture of edible films with pectin extracted from mango peel. The effect of mixing temperature, pectin concentration, and type of plasticizer on the characteristics of edible films was investigated in more depth. The characteristics of edible films are measured from thickness, tensile strength, elongation, and solubility.

2. Method

2.1. Tools and Materials

The research equipment includes aerator, blender, porcelain dish, 10x15 cm mold, glass funnel, desiccator, bucket, beaker, measuring cup, hotplate, caliper, watch glass, spiral condenser, three neck flask, measuring flask, magnetic stirrer, balance analytics, oven, water bath, ruler, volume pipette, 40 mesh screener, hose, stopwatch, alcohol thermometer and Universal Testing Machine test kit. The materials used in this study consisted of aluminum foil, aqua distillate, oxalic acid, 96% ethanol, glycerol, filter paper, mango peel, tapioca flour, polyethylene glycol (PEG 400), and sorbitol. The schematic of the main equipment set is shown in Fig. 1.



Remarks:
1. Static
2. Beaker Glass
3. Thermometer
4. Magnetic Stirrer
5. Hot Plate

Fig. 1. The main equipment set for manufacturing edible film.

2.2. Extraction of Pectin from Mango Peel

The extraction of pectin from mango peel is described schematically in Fig. 2.

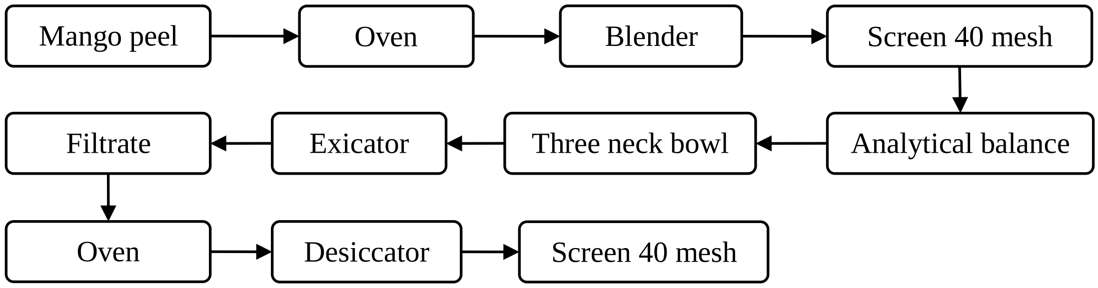


Fig. 2. Scheme of extraction of pectin from mango peel.

2.3. Manufacture of Edible Film

The procedure of manufacture of edible film is presented in Fig. 3.

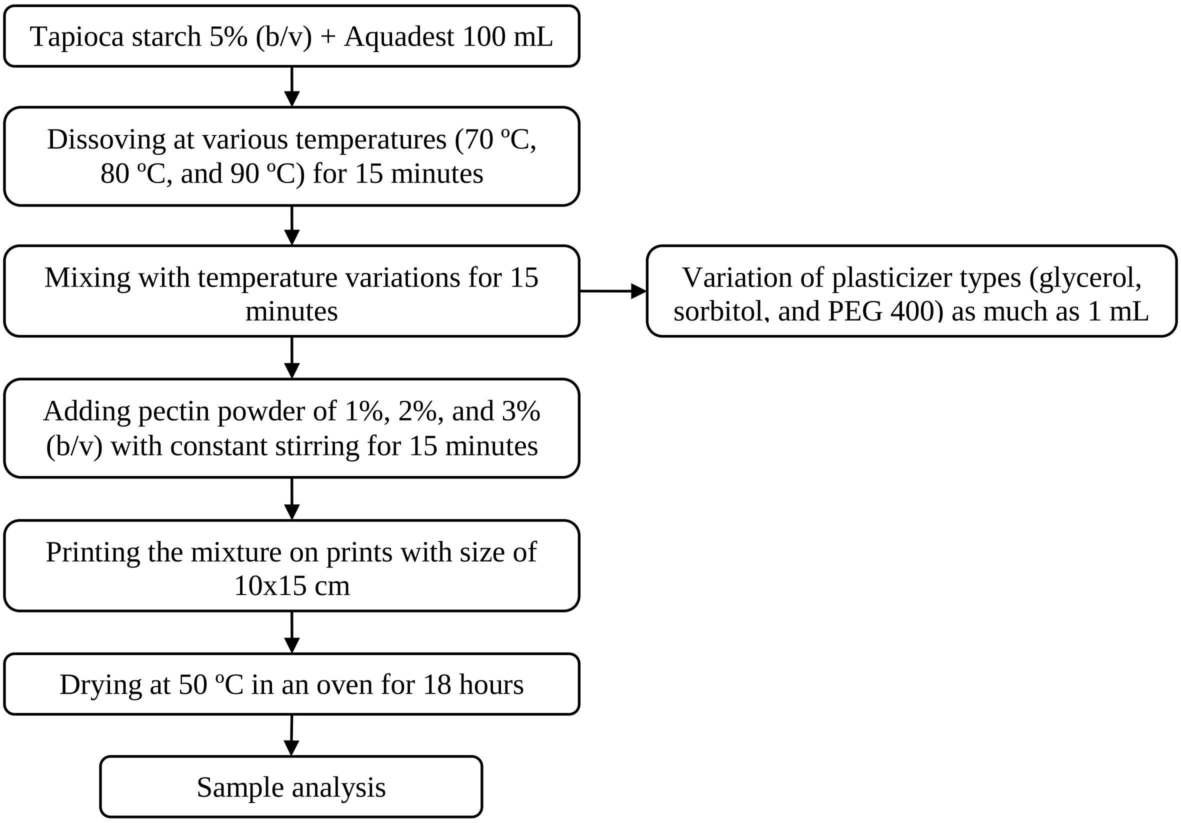


Fig. 3. Schematic procedure of manufacture of edible film.

Tapioca starch 5% (w/v) was dissolved in 100 mL of distilled water. The solution was stirred until the starch was dissolved at various temperatures (70 ºC, 80 ºC, and 90 ºC) with a constant stirring speed of 500 rpm for 15 minutes. One mL of various types of plasticizers (glycerol, sorbitol, and PEG 400) were added to the starch solution, with heating and stirring kept constant for 15 minutes. Pectin powder 1%, 2%, and 3% (w/v) were added to the starch plasticizer solution. Heating and stirring were kept constant for 15 minutes. The homogeneous edible film solution was then stirred with a stirring rod for 2 minutes to remove bubbles. The solution was poured into a 10x15 cm mold and allowed to stand for 4 hours so that the surface of the edible film was not damaged when transferred to the oven. The edible film solution was dried at 50 ºC for 18 hours until the edible film was perfectly dry and easy to remove from the mold. The finished edible film is stored in a tightly closed container at room temperature or in a desiccator to then be tested for physical properties.

2.4. Sample Analysis

The characteristics of edible films tested were thickness, tensile strength, elongation, and solubility.

1) Thickness test

The thickness of the edible film was measured using a caliper (accuracy 0.1 mm). Measurements were made at five different points. The measurement results were then entered in the equation below.

$$\text{Thickness} = \frac{A1+A2+A3+A4+A5}{5} \tag{1}$$

2) Tensile strength and elongation test

The edible film sample was clamped with a pulling accessory on the instrument's Universal Testing Machine, then displacement was activated to stretch the sample and was stopped when the sample was disconnected. Elongation was obtained from the elongation value of the film which was the ratio of the initial length to the final length of the film at break [12]. The value of tensile strength and elongation can be determined by the following formula.

$$\text{Tensile strength}\left(\frac{N}{mm^2}\right)=\frac{\text{Force}(N)}{\text{Surface area}(mm^2)} \quad (2)$$

$$\text{Elongation}=\frac{\text{Edible film extension }(cm)}{\text{initial length of edible film}(cm)} \quad (3)$$

3) Solubility test

Edible film samples were cut to a uniform size and weighed on an analytical balance as initial weight (w_0). The porcelain cup was cleaned and oven-baked for 30 minutes at 100 °C. The sample was placed in a dry porcelain dish by adding 100 mL of distilled water, then allowed to stand for 18 hours. The undissolved sample was dried in an oven at 100 °C for 2 hours and left in a desiccator for 15 minutes. The sample was weighed on the analytical balance as the final weight (w_1). The solubility of edible films can be determined by the following equation.

$$\text{Solubility}=\frac{w_0-w_1}{w_0} \quad (4)$$

3. Results and Discussion

3.1. Effect of Temperature

The temperature greatly affects the physical and mechanical properties of edible films. This is related to the gelatinization process of mango peel pectin [13]. The difference in the mixing temperature of the ingredients that make up the edible film affects the thickness, tensile strength, elongation, and solubility of the film. The effect of mixing temperature on these characteristics is shown in Fig. 4.

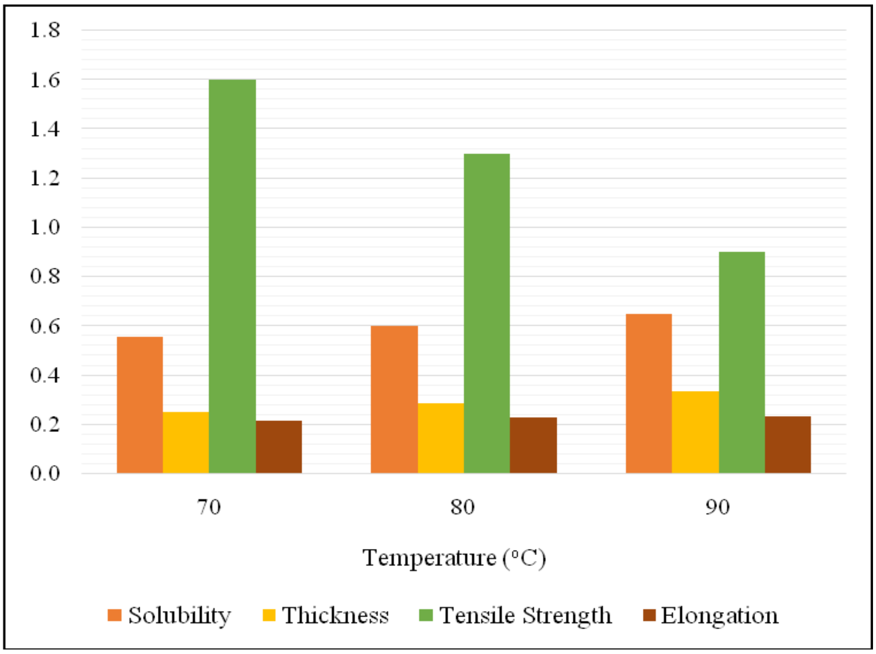


Fig. 4. Effect of temperature on the characteristics of edible film.

The thickness of edible film affects the physical and mechanical properties of edible films. The thickness was tested by measuring the edible film sheet on five different sides. It can be seen that the higher the mixing temperature, the thicker the edible film produced. The thickness obtained at temperatures of 70 °C, 80 °C, and 90 °C were 0.252 mm, 0.288 mm, and 0.336 mm, respectively. To form a gel edible film, the starch must reach its gelatinization temperature, and the gelatinization temperature of tapioca starch is 69.56 ºC [13]. Increasing the heating temperature causes the gelatinization process to be faster and more optimal so that the resulting short chain amylose fraction increases. Amylose then forms hydrogen bonds with water. The more amylose that binds, the thicker the edible film matrix.

Fig.4 shows that the higher the mixing temperature, the lower the tensile strength of the edible film obtained. The high temperature resulted in the depolymerization process of starch molecules which resulted in the short-chain amylose fraction so that the molecular weight was low [18]. Because of this process, apart from making the edible film easily soluble in water, it can also cause the edible film to have a low tensile strength value. In the mixing temperature range of 70-90 ºC, the tensile strength of the edible film decreases from 1.6 MPa to 0.9 MPa.

For elongation, the higher the mixing temperature, the higher the elongation obtained. In the temperature range of 70-90 ºC, the elongation of the edible film is 0.217-0.234. The higher the temperature used, the lower the water content in the edible film, as a result, the strength of the water molecules in the edible film decreases, and the mobility between the molecular chains increases so that the edible film is more flexible [22].

Solubility is a factor that affects the level of biodegradability of edible films when used as packaging for food products. Edible films with high solubility allow the degradation process to take place quickly but can shorten the packaging time of food. Fig. 4 shows that the solubility of the edible film increases as the mixing temperature increases. The solubility of edible film in water at 70 ºC, 80 ºC, and 90 ºC respectively was 0.556 (g water/g edible film), 0.599, and 0.648. These results are in accordance with other research, that starch suspension heated to its gelatinization temperature will be gelatinized [18]. If the heating temperature is increased, the amylose in starch will be depolymerized to produce short-chain amylose which has a low molecular weight and is easy to dissolve. This increase in the low chain amylose fraction causes an increase in the solubility of the edible film [18].

3.2. Effect of Pectin Concentration

Edible films made from starch are brittle and have low resistance. The addition of pectin can improve the physical and mechanical properties of edible film because it has a good gel formation ability [5]. The effect of the concentration of pectin used on the characteristics of the edible film is shown in Fig. 5.

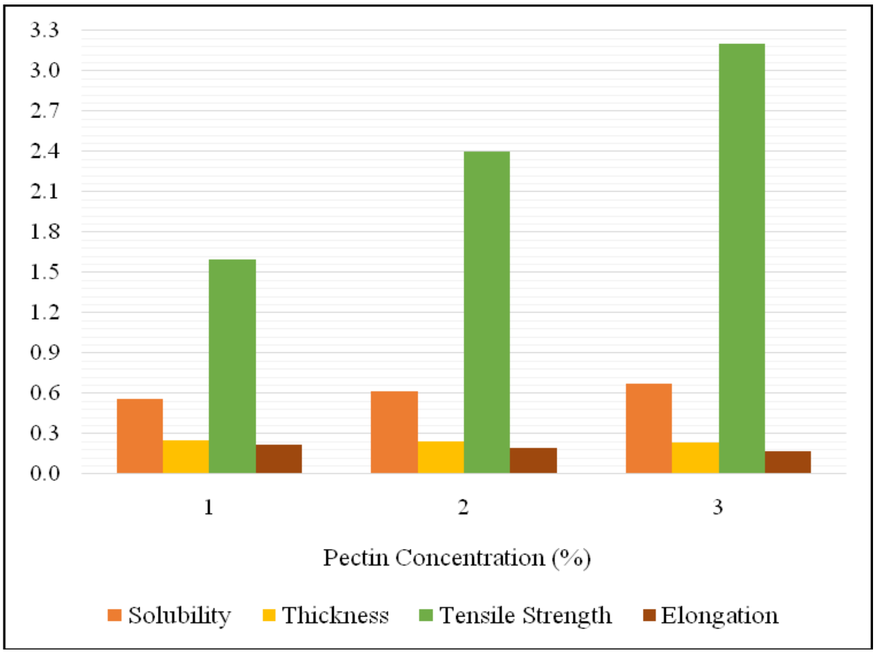


Fig. 5. The effect of pectin concentration on the characteristics of edible film.

Pectin concentration affects the thickness of the edible film. From Fig. 5 it is known that the increase in the thickness of the edible film is in line with the increase in the pectin concentration.

The thickness obtained at 1%, 2%, and 3% pectin concentration was 0.252 mm, 0.332 mm, and 0.392 mm, respectively. In general, an increase in the concentration of the material used will cause an increase in film thickness [11]. This is due to the increase in the pectin concentration will increase the number of dissolved solids, increase the viscosity of the solution, and increase the bonds between molecules so that the edible film matrix becomes denser and its thickness increases.

The larger the tensile strength of the edible film, the greater the force required to pull the film [14]. As well as its effect on the thickness of the edible film, the increase in the pectin concentration increases the tensile strength of the edible film. This result is in accordance with other research, which states that the tensile strength is directly proportional to the concentration of pectin used [19]. The higher the concentration of pectin, the interaction between the molecular matrix on the edible film will be stronger so that the tensile strength value obtained increases. In the range of 1-3% pectin concentration, the tensile strength of edible film increased from 1.6 MPa to 3.2 MPa.

Elongation of the edible film is also affected by pectin concentration, but inversely with the effect of temperature (Fig. 4). The higher the pectin concentration, the lower the elongation obtained [23]. This is because the increase in pectin will increase the number of solids in the edible film and strengthen the tensile force of the polymer so that the bonds formed are tighter and more compact which causes the edible film to be strong and difficult to elongate. In the variation of pectin concentration 1-3%, the elongation of the edible film decreased from 0.217 to 0.134.

The solubility of edible films in water is directly proportional to the pectin concentration. In the range of 1-3% pectin concentration, the solubility of the edible film ranges from 0.556 to 0.670. Pectin is a hydrophilic component that easily interacts with water molecules. There is a large influence of the physical properties of edible films on solubility, namely the more hydrophobic components, the lower the solubility; conversely, the more hydrophilic components will increase the solubility of the edible film [24].

3.3. Effect of Type of Plasticizer

The addition of plasticizers is intended to improve the physical and mechanical properties of edible films. The polymer formed from gelatinization of starch and pectin has a weakness in its stiffness. Plasticizer serves to weaken the rigidity of the polymer and provide flexible properties to the film [17]. The plasticizer used were glycerol, sorbitol, and PEG 400. The effect of the type of plasticizer on the characteristics of the edible film is shown in Fig. 6.

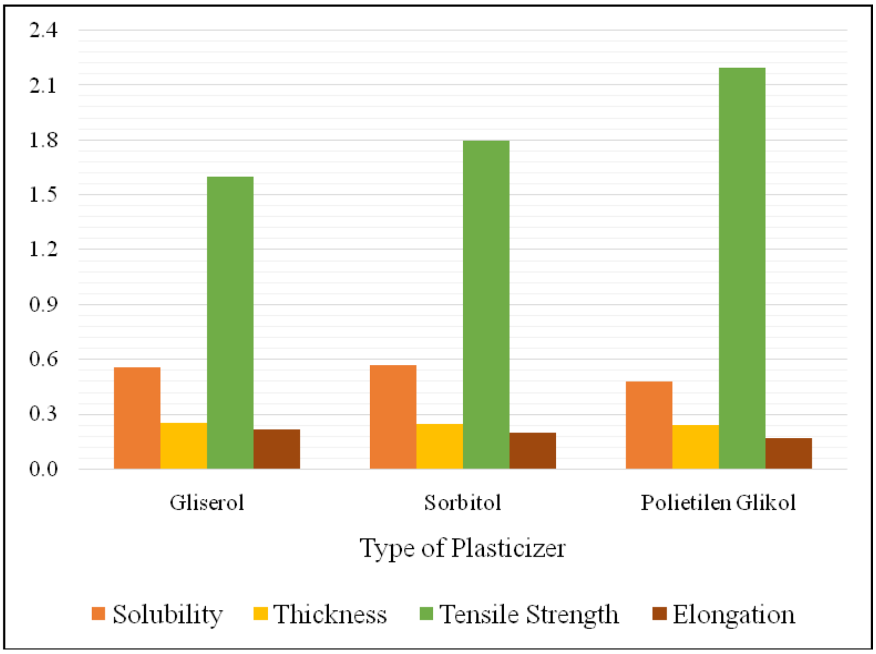


Fig. 6. The effect of type of plasticizer on the characteristics of edible film.

Fig. 6 shows that there are differences in thickness values caused by the type of plasticizer used. This difference is based on the ability to absorb water for each type of plasticizer. The easier the plasticizer absorbs water, the more water content in the edible film increases so that its thickness increases [15]. The hygroscopic ability of glycerol and sorbitol is higher than PEG 400 which is known to only have a static hygroscopicity of 60% of the static hygroscopicity of glycerol.

The tensile strength of the 3 plasticizers has different values. The tensile strength of edible films using PEG 400 is higher than that of glycerol and sorbitol. The tensile strength value obtained for the glycerol plasticizer is 1.6 MPa, the sorbitol plasticizer is 1.8 MPa, and the PEG 400 plasticizer is 2.2 MPa. In this case, the molecular weight of the material affects the strength of the edible film. The low molecular weight of the material causes the space between polymers to increase, thereby reducing the tensile strength value [20]. The molecular weights from the lowest were plasticizer glycerol (92 g/mol), sorbitol (182 g/mol), and PEG 400 (400 g/mol).

The type of plasticizer affects the elongation of the edible film. Edible films that use glycerol as a plasticizer are more elastic than sorbitol and PEG 400. Glycerol is a liquid phase at room temperature, while sorbitol plasticizers tend to be crystalline [17]. The crystallization of sorbitol reduces the flexibility of the edible film. The manufacture of edible films using the plasticizer PEG 400 resulted in a lower elongation value when compared to the other two plasticizers due to the large molecular weight of PEG 400 which reduced the space between polymers so that their elongation decreased [12,15].

The solubility of edible films is also influenced by the type of plasticizer. The solubility of edible film obtained in the glycerol plasticizer is 0.556, the sorbitol plasticizer is 0.567, and the PEG 400 plasticizer is 0.477. This difference is influenced by the ability to bind water molecules of each plasticizer. Glycerol and sorbitol have very hygroscopic properties so that it is easier to absorb water molecules compared to PEG 400 [15].

3.4. Results Comparison

The average value of the thickness of the edible film in nine different treatments was 0.287 mm. This value is quite high when compared to several similar studies. The thickness resulting from the addition of banana peel pectin 1-3% (w/v) ranged from 0.059-0.125 mm [23]. The difference is mainly due to the high concentration of starch used, as much as 5% (w/v). Based on Japanese Industrial Standard data, the standard thickness for food wrapping film is 0.25 mm [16]. The average thickness produced is good because it has exceeded the standard value. To obtain the highest thickness edible film, it is recommended to manufacture the edible film with the addition of 3% pectin, a mixing temperature of 90 ^oC, and use a plasticizer of glycerol.

The average tensile strength of edible film from nine different treatments was 1.844 MPa. The tensile strength obtained is quite small when compared to the study of edible film from durian peel pectin. The tensile strength of edible film made from durian peel pectin at a concentration variation of 0-10% (w/v) is 4.727 MPa which is caused by more pectin used in film making [5]. The standard value for tensile strength for plastics based on the Indonesian National Standard is 24.07-302 MPa [21]. The results of the tensile strength test of the edible film have a lower value than the SNI value. From the whole research results due to the variations of pectin concentration, mixing temperature, and type of plasticizer, the recommended operating conditions for making edible films to obtain optimal tensile strength is by adding a pectin concentration of 3%, using PEG 400 as plasticizer with a mixing temperature of 70 ^oC.

The average elongation of edible films obtained from nine different variations is 0.2, while the standard elongation value for plastics based on Indonesian National Standard data is 0.21–2.2 [21]. The results for the elongation test are good, although there are some variations that have a lower value when compared to the SNI. The results obtained are in accordance with several studies that have been carried out. The elongation of the edible film decreases in line with the increased stiffness of the film from the process of increasing the concentration of pectin. The elongation of the edible film ranges from 0.09 to 0.154 [23]. From the whole observation, to obtain optimal elongation, it is recommended to add a pectin concentration of 1%, using glycerol as a plasticizer with a mixing temperature of 70 ^oC.

The average solubility of the edible film obtained from nine different variations is 0.582. This is in accordance with a similar study which showed that there was a difference in the effect of adding durian peel pectin with a concentration variation of 0-10% (w/v) on the solubility of the film. The solubility of the durian skin edible film which is soluble in water is 0.447 [5]. The solubility obtained is good because it is not much different from the solubility of similar studies. Of the nine variations that have been tested, it can be recommended compositions to obtain optimal solubility values, namely, mango peel pectin with a concentration of 3% with sorbitol plasticizer and a mixing temperature of 90 ^{o}C.

4. Conclusion

The manufacture of edible films with the addition of pectin extracted from mango peel has been carried out. The mixing temperature, pectin concentration, and the type of plasticizer are known to affect the characteristics of the edible film, including thickness, tensile strength, elongation, and solubility. The higher the mixing temperature increases the solubility, thickness, and elongation values, but decreases the tensile strength. Increasing the concentration of pectin increases solubility, thickness, and tensile strength, but decreases elongation. The use of glycerol plasticizer is better when compared to the use of sorbitol plasticizer and PEG 400 plasticizer. In the studied variable intervals, the most optimal mixing temperature conditions and pectin concentration resulted in the best edible film characteristics being 70 ^{o}C and 3%, giving the solubility of 0.670, the thickness of 0.392 mm, tensile strength of 3,200 MPa, and elongation of 0.134.

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