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Characterization Bio-Based Edible Film from Mango Seed Starch and Semi-Refined Carrageenan (*Euchema cottonii*) Using Sorbitol Plasticizer for Potential Food Contact Materials

Sintha Soraya Santi[1*], Ika Nawang Puspitawati[1], Tim Pasang[1]

[1]Department of Chemical Engineering, Faculty of Engineering and Science, Universitas Pembangunan Nasional Veteran Jawa Timur, Surabaya, Indonesia.

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Corresponding Author:
Sintha Soraya Santi
sintha.tk@upnjatim.ac.id

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Abstract: Meanwhile, Semi-Refined Carrageenan (SRC) could be combined with starch as the base material for edible film fabrication to increase its tensile strength. This study aimed to identify edible film by synthesizing SRC and mango seed starch with plasticizer sorbitol, which could be safe for consumption in food packaging. The process of making edible film consists of three steps: extracting *Eucheuma cottonii* seaweed, making starch from mango seeds, and making edible film. The method used was the solution casting method, with a ratio of SRC: starch of mango seeds, namely 1: 0; 0.7: 0.3; and 0: 1 (w / w) and sorbitol concentrations of 20, 30, 40, 50, and 60% (w/w). This research shows that increasing sorbitol could decrease the tensile strength but increase the elongation and solubility of the edible film. As well as the combination of SRC and mango seed starch can produce edible films with higher characteristics than edible films based only on SRC or mango seed starch. The results of selecting the best conditions for an edible film based on the Japanese Industrial Standard (JIS) are the concentration ratio of SRC: mango seed starch (0.7: 0.3) and 30% sorbitol concentration with a thickness value of 0.22 mm, tensile strength 4.81 MPa, 28.50% elongation and 68.44% solubility.

Keywords: Characterization; Edible film; Elongation; Mango seed starch; Semi-Refined Carrageenan (SRC)

Introduction

Food contact materials (FCMs) come into contact with food, including packaging, containers, utensils, and plates. They play a crucial role in the entire food industry, from production to distribution. Currently, FCMs predominantly consist of plastic, rubber, paper, and metal. Microplastics and toxic additives from plastics can enter the food chain (Perera et al., 2021). Environmental concerns are increasingly focused on developing biodegradable films from natural sources as an alternative to petrochemical-based plastics. Researchers have been working on creating edible and degradable food films using natural and renewable polymers like polysaccharides, proteins, and lipids. These natural biopolymers offer edibility and degradability and exhibit biocompatibility with other functional materials, providing numerous advantages in food packaging (Hamid et al., 2018). The abundant algae production in Indonesia presents an opportunity for the carrageenan industry. The production volume reached 1.7 million tons, which gradually rose in the following years, reaching 5.1 million tons in 2011. The processing of carrageenan into edible films is expected to drive the growth of the carrageenan industry (Manuhara et al., 2016).

Carrageenan, a natural polysaccharide polymer obtained from red seaweed of the Rhodophyceae family, has been recognized as a potential biopolymer capable of forming solid gels and providing practical barriers against gases, fats, and oils (Puscaselu et al., 2020). Carrageenan is classified into two purity levels: refined carrageenan (RC) and semi-refined carrageenan (SRC). SRC has a lower purity level than RC due to a small

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amount of cellulose that settles with carrageenan (Ganesan et al., 2019). As an alternative, SRC has proven to be a viable option for producing food packaging films due to its lower cost (Hamid et al., 2019). Innovations in blending polymers with active compounds have led to the development of new film materials with desired functional properties, offering specific protection for food. Semi-refined carrageenan (SRC), a resource for bio-edible film packaging, typically involves natural polymers (Sedayu et al., 2020). This study used mango seeds, which are a substitute for staple foods.

Mango seed starch has an amylose content of 35.32% and amylopectin of 45.98% (Bangar et al., 2021). The high starch content of mango seeds can be used as raw material for making edible films (Torres-León et al., 2018). Starch is a polysaccharide from plants that is abundant in nature, biodegradable, easy to obtain, and inexpensive (Khalil et al., 2017). The ratio between SRC concentration with mango seed starch and sorbitol plasticizer concentration can affect the characteristics of the edible film, such as tensile strength and elongation value (Sanyang et al., 2015). The addition of sorbitol as a plasticizer makes the resulting edible film elastic (Martins et al., 2022). Using less than 1% carrageenan produces a very dilute solution and forms a thin film, making it difficult to remove from the mold and tearing easily. In comparison, using more than 3% carrageenan produces a viscous solution and forms films with uneven thickness (Mathew et al., 2024; Setyorini & Nurcahyani, 2016). Using 2% carrageenan has a less runny and thick resolution, making printing easier (Zia et al., 2017). Recent research has shown that starch-based bioplastics have a weakness: their rigid nature and low tensile strength and elasticity values.

Therefore, plasticizers and hydrophilic materials are required (Bulatovic et al., 2021). Plasticizer agent(s) are added to improve their elasticity (Eslami et al., 2023). According to research by Suderman et al. (2018) and Ballesteros-Mártinez et al. (2020), the use of sorbitol as a plasticizer has a higher tensile strength and elongation value compared to the use of glycerol (Paudel et al., 2023). Based on these parameters, research entitled "Synthesis of Edible Film from Mango Seed Starch and Semi-Refined Carrageenan *(Euchema Cottonii*) with Sorbitol Plasticizer for Potential Food Contact Materials was carried out. The combination of Semirefined Carrageenan (SRC) and mango seed starch has not been used as a raw material for making edible films with sorbitol plasticizer, so we can learn about the characteristics of edible film.

Method

Materials

Materials used in this study were Semirefined Carrageenan extracted from *Euchema cotton* seaweed obtained from coastal seaweed farmers in Pamekasan – East Java, starch from mango seed waste obtained from household waste and home industry, sorbitol to improve the mechanical quality of edible films purchased from CV Nura Jaya Surabaya, KOH to reduce the amount of sulfate in Carrageenan purchased from UD Saba Kimia Surabaya, and Aquadest as a solvent purchased from UD Saba Kimia Surabaya.

Preparation of Semirefined Carrageenan (SRC)

Dried *Euchema cottoni* seaweed cut into small pieces. Seaweed was put into a beaker glass, then 8% KOH solution was (with a ratio of 1 6 for seaweed: 8% KOH) and heated at 80^{o}C for 2 hours. After heating, let it stand for 30 minutes until the temperature is the same as the room temperature. Seaweed was filtered with a filter cloth and washed with water to reduce excess KOH. The seaweed is dried and then ground to a powder.

Preparation of Starch from Mango Seeds

The spermodermis cleans the mango seeds. Cut into small pieces. Crushed using a blender with the help of water. Squeezed through a sieve into a container until the dregs no longer drank juice. Let it settle for up to 24 hours. The resulting suspension is decanted. The resulting starch is then dried by drying it under the hot sun. Then smoothed with a mortar.

Edible Film Production

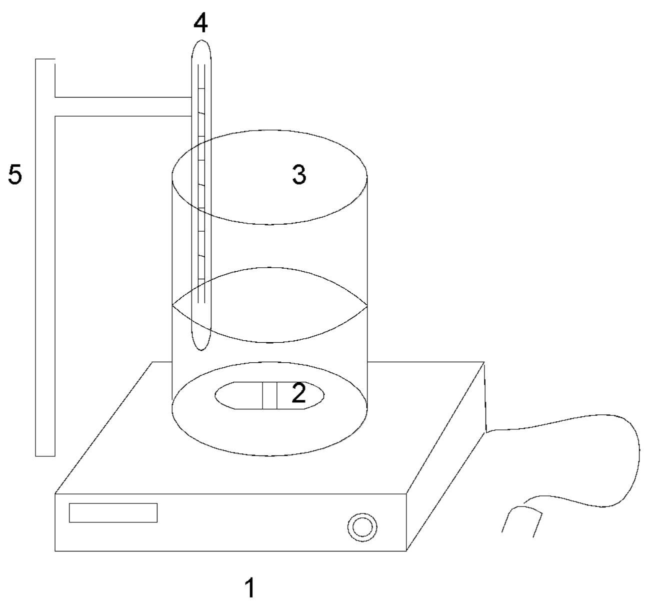

Figure 1. Edible Film from the mold

Information: Hot Plate Magnetic Stirrer (1), Magnetic Capsules (2), Beaker Glass (3), Thermometer (4), Statives and Clamps (5).

In the initial stage, namely weighing SRC and mango seed starch with a total solid of 2 grams, the weight ratio of SRC and mango seed starch is 1:0; 0.7:0.3; 0:1 (w/w). Stirred the SRC solution and mango seed flour with 100 ml of hot distilled water for ± 5 minutes until homogeneous. Next, sorbitol was added to the mixture of SRC and mango seed starch with variations of 20, 30, 40, 50, and 60% of the dry weight of the ingredients. Stir again using a magnetic stirrer for 30 minutes at 80°C until homogeneous. It cooled the Edible Film Solution to room temperature and dry at 65°C for 24 hours. In the final stage, dry the Edible Film at room temperature until it cools, then remove the Edible Film from the mold.

Edible Film Thickness Measurement

The resulting thickness was measured using a micrometer with an accuracy of 0.0001 mm. Measurements were taken at five different places to get the average thickness (Moey et al., 2014). Determination of Mechanical Properties. The tensile test was carried out with both ends of the sample clamped with an autograph. The tensile test will obtain information regarding the tensile strength and elongation of the edible film (Tafa et al., 2023).
Calculation:

$$\text{Tensile Strength(MPa)} = \sigma = \frac{F}{A \times 100} \quad (1)$$

$$\text{Elongation at Break (\%)} = \varepsilon = \frac{l - l_o}{l_o} \times 100\% \quad (2)$$

Where:
F = Tensile force (kN)
A = Area (m^2)
Δl = Elongation experienced after being stretched (cm)
l_0 = Initial elongation (cm)

Film Solubility in Water

The solubility test was carried out by weighing the weight of the initial edible film. The solubility test non instrumental measurement, A test will follow, and then the edible film into the water for 10 minutes. After 10 minutes, the edible film was removed and drained so it was not too wet and weighed again (Tafa et al., 2023).

$$\text{Solubility} = \frac{W - W_o}{W_o} \times 100\% \quad (3)$$

Where:
Wo = weight of material before soaking in water (grams)
W = weight of material after soaking in water (grams)

FTIR

FTIR spectroscopy is used to see whether a specific interaction exists between semirefined carrageenan and starch in the mixture. Analysis with the FTIR spectroscope was carried out with KBr pellets by adding 1 mg of finely powdered film to 200 mg of KBr the FTIR spectra for each sample were recorded at room temperature in the 400-4000 cm^{-1}, using 100 scans and a resolution of 4 cm^{-1} (Silva et al., 2019).

Result and Discussion

Edible Film Thickness Test Result Based on SRC Ratio: Mango Seed Starch and Sorbitol Percent

Thickness is an essential parameter because it affects the product's shelf life; if thicker, the water vapor and gas transmission rate will be lower (Adilah et al., 2018). Differences in the hydrophilic properties of carrageenan, starch, and plasticizers cause differences in thickness values. This hydrophilic property can bind more water which will evaporate after the oven process. An excellent edible film has a thickness of 0.15 - 0.20, as indicated by research conducted, as it can withstand external pressure and inhibit water vapor transmission (Battisti et al., 2017). Thickness is an important parameter that affects its use in product formation and packaging. The thickness of the film affects gas permeability.

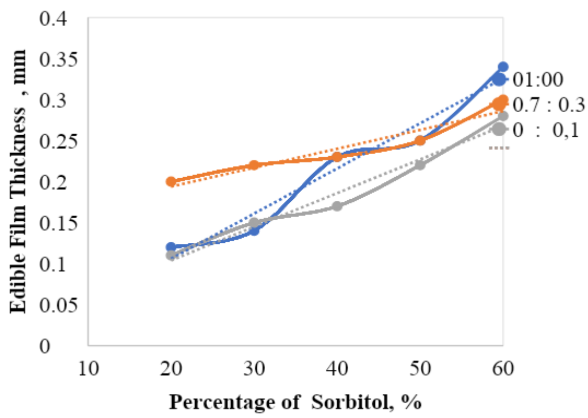

Figure 2. Graph relationship between SRC ratio: mango seed starch and sorbitol percent to edible film thickness (mm)

Thicker edible films have lower gas permeability, providing better protection for products such as sausages, meatballs, nuggets, and others that are packaged. Moreover, supported by the statement higher volume of water in the material increases the thickness of the edible film with the same surface area (Razavi et al., 2015). Therefore, the thickness of the edible film is closely related to the moisture content of the material. The higher the moisture content in the edible film, the greater its thickness. In addition to moisture content, thickness is a physical property influenced by the concentration of dissolved solids in the film solution (Hamsina et al., 2024).

Result of Tensile Strength (MPa) Based on SRC Ratio: Mango Seed Starch and Percent Sorbitol

Tensile strength is an edible film's mechanical property, which is crucial because it is related to the ability of edible film to protect the coated product. Tensile strength is the maximum stress a film can withstand before breaking. An edible film with high tensile strength is required for food product packaging, which aims to protect food ingredients during handling, transportation, and marketing. The increase in attractive forces between the molecules composing the edible film enhances its structural strength (Balqis et al., 2017; Han, 2014).

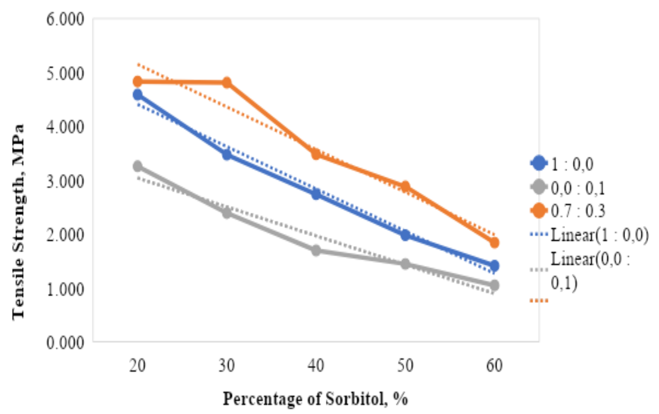

Figure 3. Graph relationship between SRC ratio: mango seed starch and percent of sorbitol to edible films tensile strength (MPa)

Figure 3 shows the tensile strength results obtained when the ratio of SRC: mango seed starch (1:0; 0.7:0.3 and 0:1) decreased with increasing sorbitol concentration. It happens because adding a plasticizer can reduce intermolecular bonds between polymer chains, reducing the film structure's stiffness and improving the polymer's mobility. In addition, the plasticizer can reduce the tensile strength value because the bonds between polysaccharides that are broken by the plasticizer cause space, so the bonds between molecules in the film weaken (Cabello et al., 2015; Rezaei & Motamedzadegan, 2015). These results are supported by research from Liang & Wang (2018) and Zhang et al. (2016). The results of the study also showed that at each addition of sorbitol concentration, the tensile strength in the ratio of SRC: mango seed starch (0.7:0.3) was higher than the ratio of SRC: mango seed starch (1:0 and 0:1), that is, from 1.840 MPa – 4.831 MPa. It shows that the combination of SRC and mango seed starch can produce edible films with higher tensile strength than those based on SRC or mango seed starch. These results show that edible films function to increase the density of the mixture which can cause regular arrangement in the molecule thereby increasing the tensile strength value of the edible film (Bharti et al., 2021).

In the sorbitol variation, the tensile strength value of the SRC: mango seed starch ratio (1:0) was higher than the SRC: mango seed starch ratio (0:1). This is because carrageenan can form a strong polymer matrix, where carrageenan will dissolve in each polymer chain and fill all spaces thereby reducing molecular motion and making intermolecular tensile strength stronger on edible films (Sogut et al., 2019). The highest tensile strength value results in the ratio of SRC: Mango seed starch (0.7: 0.3) with 20% sorbitol, equal to 4.831 MPa. Meanwhile, for the lowest percent elongation in the SRC – Mango seed starch (0: 1) ratio with 60% sorbitol, equal to 1.047 MPa. Overall, three composition variations meet the minimum standard for the tensile strength of edible films based on the Japanese Industrial Standard (JIS) of 3.92 MPa, namely the ratio of SRC: Starch of mango seeds (0.7: 0.3) with 20% sorbitol and 30%, that is equal to 4.831 and 4.810 MPa. As well as on the ratio SRC: Mango seed starch (1: 0) with 20% sorbitol, with a tensile strength value of 4.588 MPa.

The R^2 value in Figure 3 interprets the relationship between the thickness of the edible film with the addition of sorbitol concentration and the SRC ratio of Mango seed starch. The R^2 value of 0.9855, 0.9478, 0.9478 respectively indicates that the tensile strength of edible film is greatly influenced by the addition of sorbitol concentration to the SRC and Mango seed starch ratio.

The Result of the Elongation Test (%) Based on the SRC Ratio: Mango Seed Starch and Sorbitol Percent

Elongation is the percentage change in the film's length, calculated when the film is stretched to break. The elongation value is generally inversely related to the tensile strength value. Figure 4 shows the elongation results obtained when the ratio of SRC: mango seed starch (1:0; 0.7:0.3, and 0:1) increased with increasing sorbitol concentration. Polymer chains reduce the stiffness of the film structure and increase polymer mobility. Increasing the plasticizer concentration will cause a decrease in internal hydrogen bonds so that the interaction of intermolecular and intramolecular hydrogen bonds of adjacent polymer chains will be weaker, and the film will be more flexible so that the percent elongation value will increase (Esmaeili et al., 2017).

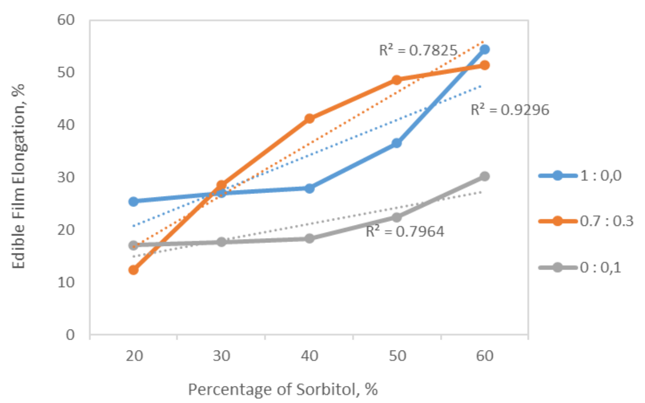

Figure 4. Graph relationship between SRC ratio: mango seed starch and sorbitol percent on edible film elongation (%)

At 30% sorbitol concentration, the elongation ratio of SRC: mango seed starch (0.7:0.3) was higher than the SRC: mango seed starch ratio (1:0 and 0:1), namely 28.50%. The same thing happened at sorbitol concentrations of 40 and 50%; elongation in the percentage of SRC: mango seed starch (0.7:0.3) was higher than the ratio of SRC: mango seed starch (1:0 and 0:1), namely 41.20 and 48.60%. It shows that the combination of SRC and mango seed starch can produce edible films with higher elongation than those based only on SRC or mango seed starch alone. In the sorbitol variation, the elongation value of the SRC: mango seed starch ratio (1:0) was higher than the SRC: mango seed starch ratio (0:1). This is because SRC can bind water better to produce a gel matrix that can increase the percent elongation of edible films.

The highest percent elongation results were found in the SRC: Mango seed starch (1:0) ratio with 60% sorbitol, which was 54.40%. Meanwhile, the lowest percent elongation was in the ratio of SRC – Mango seed starch (0.7: 0.3) with 20% sorbitol, which was 12.40%. Overall, the percent elongation of edible films produced in this study met the minimum standard for the percent elongation of edible films based on the Japanese Industrial Standard, namely 10%. Elongation is the percentage change in the film's length, calculated when the film is stretched to break. The elongation value is generally inversely related to the tensile strength value (Zhang et al., 2022; Shiraiwa et al., 2022).

The Result of the Solubility Test (%) Based on the SRC Ratio: Mango Seed Starch and Percent of Sorbitol

Solubility is a physical property of an edible film that shows the percentage of dissolved dry weight after being dipped in water. The edible film with high solubility means its ability to hold water is reduced but can be easily consumed, revealing that high solubility is required if a movie is applied to food packaging with low water content. Vice versa, if a film wants to be involved as food packaging with a high water content, it requires low solubility (Al-Hassan & Norziah, 2012; Thakur et al., 2019).

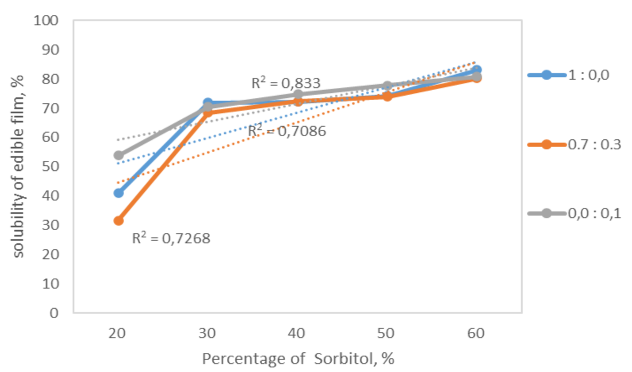

Figure 5. Graph relationship between SRC ratio: mango seed starch and sorbitol percent solubility (%) of edible films

Figure 5 shows that the solubility of edible films increases with increasing sorbitol concentration. It was because sorbitol has a strong affinity for water molecules and a low molecular weight which can help sorbitol enter between polymer chains, thereby increasing the free space between polymer chains. In addition, sorbitol also has hydrophilic properties, which can increase the solubility of edible films compared to materials that have hydrophobic properties. The highest percent solubility was obtained at the ratio of SRC: Mango seed starch (1: 0) with 60% sorbitol, which was 83.105%. While the lowest yield was obtained at the ratio of SRC – Mango seed starch (0.7: 0.3) with 20% sorbitol, which is 31.63%.

Result of FTIR Test

Figure 6 shows the presence of CO at wave numbers 1050-1300 cm^{-1}, where this functional group is found in SRC. The addition of sorbitol as a plasticizer causes the edible film to contain OH alcohol/phenol hydrogen bond groups at wave numbers 3200-3600 cm^{-1}. The FTIR test showed that the OH group was detected at wave number 3396.51 cm^{-1}, the CH group at wave number 2937.47 cm^{-1}, and S=O (ester sulfate) at wave number 1210.85 cm^{-1}. At the same time, the CH group can be obtained from mango seed starch, SRC, and sorbitol, which have a lot of Carbon and Hydrogen elements in their chemical formula.

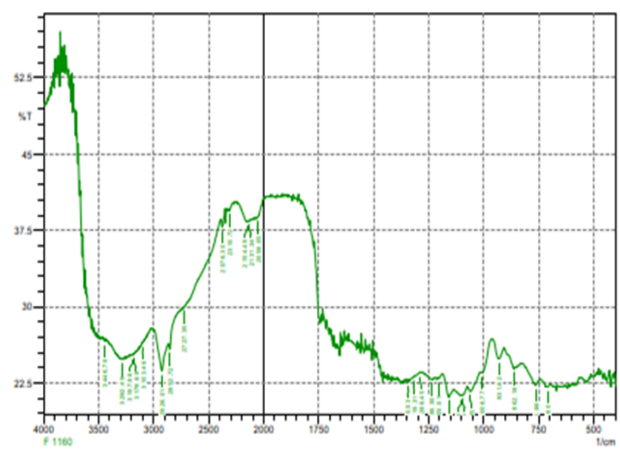

Figure 6. Result of edible films FTIR test

From the edible film FTIR test, the reading results are obtained as follows.

Table 1. Edible film FTIR test readings

Wave number (cm^{-1})	Functional groups
675-995	CH Alkenes
690-900	CH Aromatic Ring
1050-1300	CO Alcohol
2100-2260	C=C Alkyne
2850-2970	CH Alkanes
3200-3600	O H Hydrogen bonded alcohol

The Result of Physical and Mechanical Properties of Film Comparison

This comparison table will show the physical and mechanical properties of films from this research and other researchers. Characteristics of edible film, namely Thickness (mm), Solubility (%), Tensile strength (MPa), and Elongation at break (%), which previous researchers support, show figures that are still within the range or not much different.

Table 2. Physical and mechanical properties of films Comparison

Source	Thickness (mm)	Solubility (%)	Tensile strength (MPa)	Elongation at break (%)
This Research	0.11 – 0.34	31.63 – 83.10	1.84 – 4.83	41.20 – 48.60
Bulatovic et al. (2021)	-	-	4.10	5.3 – 22.30
Adilah et al. (2018)	0.39 – 0.42	23.31 – 40.06	7.65	42.09 – 59.30
Vrijens et al. (2017)	0.24 – 0.76	-	0.60– 15	37 – 62
Moey et al. (2014)	0.076	66.08	6.82	17.90

Conclusion

Producing Edible films have an average solubility of more than 60%, indicating that the SRC edible film and mango seed starch with plasticizer sorbitol is easy to dissolve and suitable for food packaging. The addition of sorbitol decreased the tensile strength but increased the elongation and solubility of the edible film. Also, the combination of SRC and mango seed starch can produce edible films with higher characteristics than those based only on SRC or just with mango seed starch. The results in selecting the best conditions for the characteristics of edible film for food contact materials by the Japanese Industrial Standard (JIS) were the ratio of SRC concentration: mango seed starch (0.7: 0.3) and 30% sorbitol concentration with a thickness value of 0.22 mm, tensile strength of 4.81 MPa, 28.50% elongation and 68.44% solubility.

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Author Contributions

Conceptualization, data curation, visualization, formal analysis, investigation, and resources, S.S.S.; methodology and writing—original draft preparation, I.N.P.; validation and writing—review, and editing, T. P. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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