THE EFFECT OF DIFFERENT PLASTICIZERS ON THE PROPERTIES OF STARCH-PROTEIN BASED EDIBLE FILM

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Abstract—**The purpose of this study is to produce edible films from starch, protein, and combination of both. Also, the aim is to investigate the effect of different types of plasticizer and their concentrations on the properties of the edible film produced as well as to compare the properties of composite edible film the one made from starch and protein. Plasticizers like glycerol, sorbitol and polyethylene glycol (PEG_{200}) were added into formulation at various concentrations (10, 15, 20, 25 %) to increase the properties of edible films. Then the combination of starch, gelatin and plasticizers were formulated and cast on petri dish to produce composite edible films and their mechanical properties (tensile strength and elongation at break, EAB), solubility and water vapor permeability (WVP) were tested. The findings showed that, the addition of gelatin into the formulation increased the tensile strength of glycerol and sorbitol plasticized film, solubility and WVP of all types of plasticized films. Besides, the increasing concentration of plasticizers will decreased the tensile strength and solubility of glycerol and sorbitol plasticized films. The EAB of the edible films increased when the concentration of sorbitol and PEG_{200} increased as well as when concentration of glycerol decreased. The increasing of concentration of plasticizers did not produce significant trend on the WVP of the composite edible films. It was observed that, the edible films plasticized with glycerol has higher EAB and higher solubility when plasticized with PEG_{200} compared to others. However, the addition of plasticizers into the formulation does not bring significant change to the WVP of the films.**

Keywords— **starch, protein, glycerol, sorbitol, polyethylene glyceol (PEG200), mechanical properties, solubility, water vapor permeability (WVP)**

I. INTRODUCTION

Nowadays, people all over the world tend to use products that are environmental friendly in order to reduce the pollution. Therefore, edible film for packaging has been developed as another option for synthetic plastic production and has received extensive global attention over the years because of their advantages over synthetic films. Sabina *et al* (2013) stated that edible film and coatings are proven to have functional properties as a barrier to solute and gasses and to extend food quality and shelf life. The main benefit of edible film compared to traditional synthetic is that they can be consumed together with the food products. Consequently, there is no waste to dispose and even if the films are not consumed, they could still contribute to the reduction of environmental pollution (Aruna *et al*., 2012).

The edible film can be produced from polysaccharides, proteins and lipids. There are various sources from these groups and they are all natural. Sources of polysaccharides are corn starch, tapioca starch, and cellulose while proteins are from animal gelatin, and whey powder. Furthermore, lipids films are made from waxes and paraffin as well as shellac resins. Composite film is the combination of starch and protein and it has a capability to enhance the strength of the film. The polysaccharides provide a minimum barrier to moisture due to its hydrophobicity properties. On the other hand, proteins generally give film with suitable optical and mechanical properties, however poor water vapor barrier and very sensitive to moisture. The composite films are believed to serve as good barrier to water vapor permeability and effective as gas barrier with improved strength (Al-Hassan & Norziah, 2012).

In order to obtain a good edible film with improved properties, several modifications have been made such as adding additives. The common additive used in making edible film is plasticizer. Its function is to improve the workability and film brittleness. This is achieved by reducing the strong intermolecular interactions between polysaccharides or protein molecules. The addition of plasticizers may alter the film mechanical resistance. Also, the type and concentration of plasticizer used have major effect on the physical, mechanical, thermal, and barrier properties of the films (Sanyang *et al*., 2015). Based on Altan *et al*., (2003), the common used of plasticizer are polyethylene glycol (PEG), glycerol (Gly), propylene glycol (PG), and sorbitol.

The objective of this study was to produce edible film that is safe for food packaging and consumer from starch, protein, and combination of both. The study also aimed to investigate the effect of different types of plasticizer and their concentrations on the properties of the edible film produced as well as to compare the properties of composite edible film the one made from starch and protein.

II. METHODOLOGY

A. Materials

Sago starch was purchased from Amcee Food Industries Sdn. Bhd and gelatin was from Halagel Products Sdn. Bhd. Glycerol used was obtained from ChemAr, sorbitol from Calbiochem and polyethylene glycol (PEG_{200}) form Carbowax. All chemicals were of analytical grade.

B. Preparation of Starch-Gelatin edible film

The procedure used in this study was adopted from Al-Hasan and Norziah (2013). The edible films were prepared by dissolving 3.6 g of mixture of sago starch and gelatin in 120 mL of distilled water. Three different ratios of starch to protein were used which are 1:1, 2:1, and 3:1. Then, different types of plasticizer were added to the film solution. For each film, the plasticizers (glycerol, sorbitol and PEG) are added at 10%, 15%, 20%, and 25% w/w of mixture of sago starch and gelatin respectively. First of all, sago starch was homogenized at 500 rpm and heated on a magnetic plate at 75°C for 15 minutes until the solution gelatinized. Then, gelatin and plasticizer was added. The homogenizing and heating process was continued for about 30 minutes. The film solutions were cooled to room temperature before being poured into 10 cm diameter petri dishes. To control the thickness of the film, the quantity of each film –forming solution poured onto the petri dish were the same which is 20 mL. Then, they were dried in ventilated oven at 40°C for 24 hours. After that, the dried solution film were peeled-off and stored it in desiccator containing silica gel with 56% (RH) at 30°C for further analysis.

C. Mechanical Properties

The mechanical properties analysis by using the method proposed by Nevena *et al*., (2010) which is tensile strength and elongation at break percentage by using a Tinius Olsen. First of all, three sample strips (20 mm x 50 mm) of each formulation are cut and clamped between tensile grips. The software used that linked with the machine is Qmat software and set measurement was set where initial grip length is 30 mm, cross head speed is 30 mm/min, extension range is 100 mm, and last but not least load cell is 50 N. Then, the tensile strength (MPa) and elongation at break (%E) were determined. The tensile strength of the film is a measure of maximal force per original cross-sectional area that the film could sustain before breaking while the elongation at break percentage measure the capacity of the film to extend before breaking. For this testing, at least three strips were used per treatment, with three replicates.

D. Solubility of the films

According to Rangel *et al*., (n.d.), the film were cut into (2 x 2 cm_2) specimens and dried in ventilated oven at at 107˚ C for 24 hours. Weight the film (W_i) and immersed in 50 mL of distilled water by using centrifuge tube. The tube was placed in incubator shaker at room temperature for 24 hours with 80 RPM.Then, the remaining pieces of film was filtered and dried at 107° C for 24 hours. After that the sample was weight (W_f) and the percentage of solubility (% SOL) was calculated using the following equation:

$$\% \text{ SOL} = \frac{\text{initial dry weight } (Wi) - \text{final dry weight } (Wf)}{\text{initial dry weight } (Wi)} \text{ x } 100$$

E. Water Vapor Permeability

The water vapor permeability of the edible film was determined by using method described by Rangel-Marron *et al*., (n.d.). The film is cut into 5 cm and placed on glass jar and was adjusted on the circumference with parafilm. The glass jar contains 5 mL of distilled water, leaving an inch air space between the water surface and the film. Then, the glass jar was stored in dessicator containing a silica gel at 33% relative humidity at 25%. The measurements are recorded over an 8 hours period with interval of 60 minutes. The water vapor transmission rate (WVTR) was then measured from the slope of regression analysis of weight loss as a function of time (g/s) and related to the water vapor permeability (WVP) according to the following formula:

$$\text{WVTR } (g/m^2s) = m_1/A$$
$$\text{WVP} = L \text{ x WVTR}/(p_1 - p_a)$$

Where m1 is the slope of the weight loss versus time (g/s), A is the exposed area, p_1 is the partial pressure of water vapor in the air, p_a is the air saturated to 33% relative humidity at 25˚C and L is the average thickness (mm)

III. RESULTS AND DISCUSSION

A. Mechanical properties of starch-protein edible films

i) Tensile Strength

Tensile strength is one of significant mechanical properties that commonly studied in packaging material. Tensile strength is the maximum tensile stress sustained by the sample during the tension test. High tensile strength is required for food packaging to protect food during handling, transportation and marketing. Effect of different types and concentrations of plasticizers on mechanical properties of different ratio of composite edible film were investigated. *Figure 1* showed the relationship between tensile strength of composite edible film with addition of different types of plasticizers (glycerol, sorbitol, and PEG_{200}) and concentration (0, 10, 15, 20, and 20%) at different ratio of sago starch-gelatin (1:1, 2:1 and 3:1).

The tensile strength of the composite edible film decrease from 4.917 to 0.27 Mpa and 13.39 to 1.937 MPa for ratio 1:1, from 3.063 to 1.4204 MPa and 8.8133 to 3.9993 MPa for ratio 2:1 as well as from 13.40333 to 1.4094 MPa for ratio 3:1 when concentration of glycerol and sorbitol increased from 10 to 25%. Muhammad et al., (2015) proposed that the possible reason for low tensile strength at high concentration of plasticizers is strong hydrogen bond between starch-protein intermolecular interactions more than starch-plasticizers attraction. Then, the plasticizers reduce the hydrogen bonding between the protein chains and increasing the spacing. Therefore, it will decrease the interaction between water molecule and composite edible film.

Furthermore, it is observed that the reduction of gelatin content from film (ratio from 1:1 to 2:1) for 10 % of glycerol and sorbitol concentration had decrease the tensile strength from 4.917 to 3.036 MPa and 13.39 to 8.8133 MPa. The present of protein in the film significantly reduce the tensile strength as a result of the interaction between hydroxyl groups between starch and protein that may reduce the interaction between starch chains. The result indicated that as the content of gelatin decreased from 1:1 to 2:1, the tensile strength increased from 2.3577 to 6.9457 MPa, 0.9897 to 4.614 MPa and 1.937 to 3.993 MPa for concentration of PEG_{200} at 15, 20 and 25%.

The results showed that, sorbitol have a good properties as a plasticizer followed by glycerol and PEG_{200}. Based on Al-Hasan and Norziah (2012), plasticizer like sorbitol are often referred as good plasticizers because it can interacts with water by hydrogen bonding due to the present of more hydroxyl groups in the molecule. Films plasticized with sorbitol presented higher intermolecular forces and showed a lower capacity to interact with water. According to Sothornvit and Krochta (2001), glycerol have smaller molecular weight as well as hygroscopic (tend to absorb moisture from air), therefore, glycerol have ability to increase the effectiveness as a plasticizer.

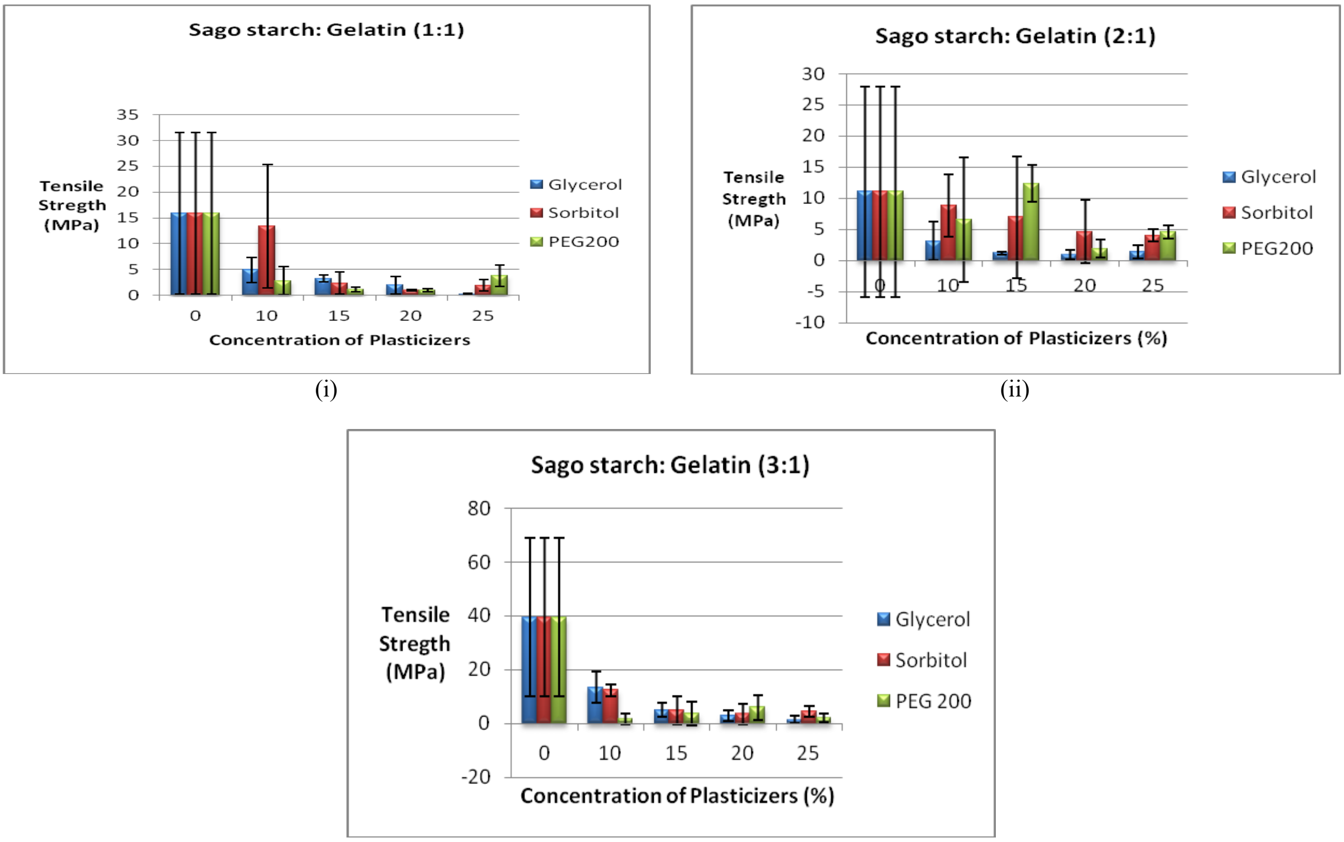

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Figure 1: Tensile Strength of edible film of different types and concentration of plasticizers at different ratio of sago starch- gelatin

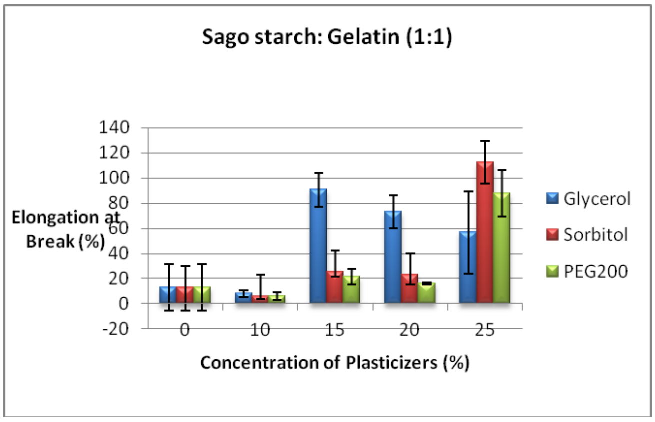
ii) Elongation at Break

Elongation at Break (EAB) of the edible film is the ability of the film to extend before break. It is related to extensibility that usually required for an edible film to stand its integrity when applied to food product. *Figure* 2 demonstrated the graph for elongation at break (EAB) for glycerol, sorbitol and polyethylene glycol 200 at 1:1, 2:1 and 3:1 ratio of sago starch-gelatin. The composite edible film produced increased in EAB from 8.3 to 56.7 %, 5.82 to 112.433% and 6.4943 to 87.9 % when the concentration of glycerol, sorbitol and PEG_{200} increased from 10 to 25% at ratio 1:1. However, the trend also similar when the gelatin content decreased to 2:1 and 3:1 ratio of sago starch-gelatin.

Based on Muhammad et al, (2015), this behavior occurred because plasticizers drop the intermolecular bonds between plasticizers and starch as well as substitute them with hydrogen bonds formed from starch and plasticizers. This interruption resulting to reduction in rigidity and encourages flexibility of films by allowing more chain mobility. The similar trends have been observed by Thawien (2013) which stated that the increasing of EAB at higher concentration of plasticizers is due to low the molecular weight hydrophilic molecules of plasticizers.

It demonstrated that the elongation at break decreased from 90.567% to 13.553% and 73.333% to 40.01% when the content of gelatin was decreased from ratio 1:1 to 3:1 for 15% and 20% of glycerol concentration. Similar relationship occurs when PEG_{200} was used as plasticizer. Al –Hassan and Norziah (2013) stated that the EAB is high when the amount of gelatin increased because gelatin seemed to act as a plastticizer which improved the edible film flexibility and reduce its brittleness.

It also showed that at 10, 15 and 20% concentration of plasticizers at ratio 1:1, the EAB of glycerol plasticized film is higher compared to sorbitol and PEG_{200} plasticized film. It concludes that glycerol could be more effective plasticizer compared to sorbitol and PEG_{200} since it is more stretchable. According to Thawiem (2013), the effectiveness of glycerol as a plasticizer in the composite edible film most likely due to its small size and configuration which allows it to be more readily inserted between the starch-protein chains. Moreover, the low molecular weight of glycerol make it is more effective as a plasticizer than those with high molecular weight.



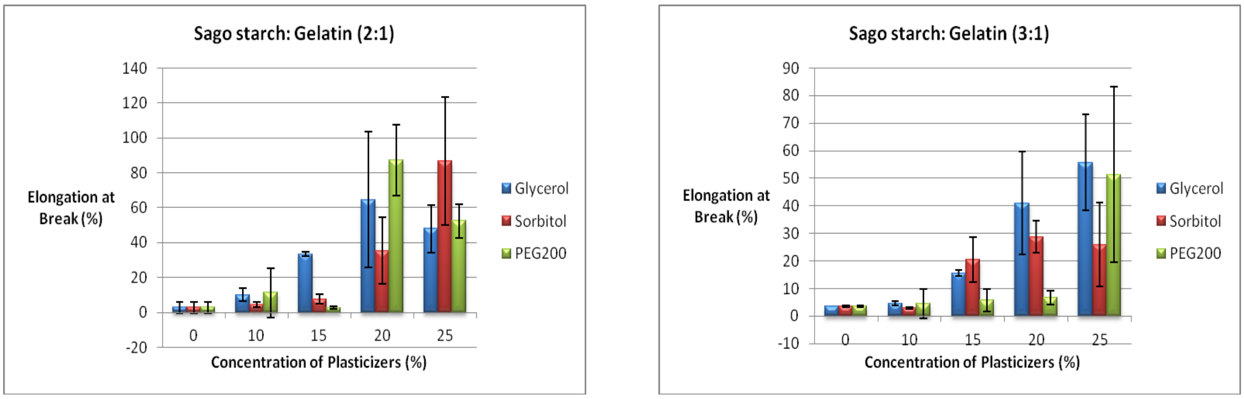


Figure 2: Elongation at Break (EAB) of edible film of different types and concentration of plasticizers at different ratio of sago starch-gelatin

B. Solubility of the films

The solubility of composite edible film in water is the major property that is related to the structural properties and the presence of components in the films. *Figure 3* provide the data for solubility test of composite edible film at different types and concentration of plasticizers and ratio of starch-gelatin. It shows that as the content of gelatin decrease from ratio 1:1 to 3:1, the solubility of the film decrease from 53.93% to 19.877%, 67.9% to 24.648% and 39.82% to 18.466% for concentrations of glycerol at 15, 20 and 25%. This correlation illustrated that the solubility of the composite edible film was decreased with decreasing the amount of gelatin content in the films. Also, it is similar occurred when sorbitol and PEG_{200} were added as plasticizers.

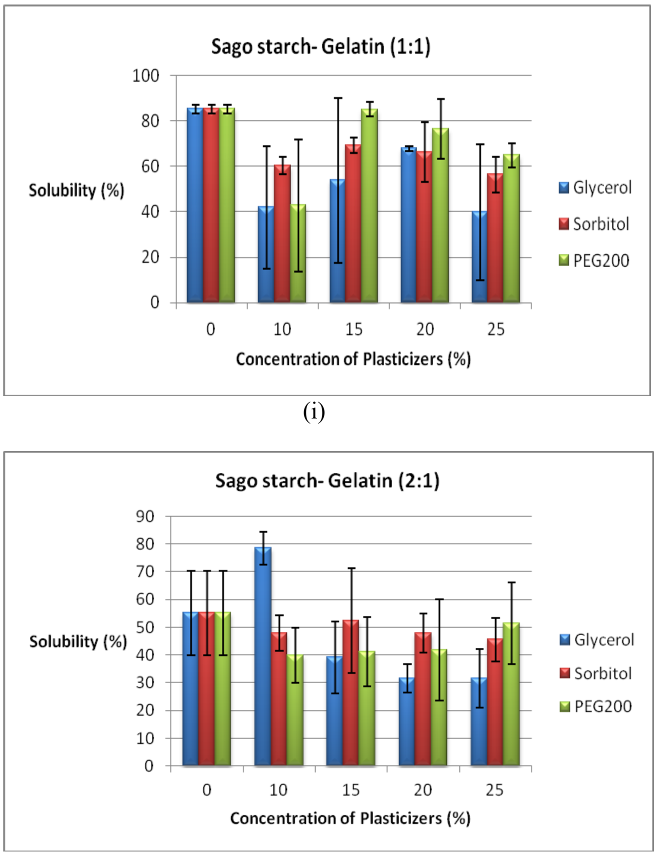
According to Farahyde et al, (2015), this trend observed maybe due to gelatin being more hydrophilic as compared to starch. Therefore, the addition of gelatin in the edible film increases the solubility of the edible films. The results showed that, the higher the amount of gelatin in edible film, the higher the solubility of composite edible film produced.

However, the result indicated that as the concentration of glycerol increase from 0 to 25%, the solubility of the film decrease from 85.25% to 39.82%, 55.123% to 31.611% and 32.094% to 18.466% for all starch-gelatin ratios. Azadeh *et al*., (2014), suggested that glycerol that interact with water and interrupts the network by hydrogen bond. The interaction of water and glycerol resulted to more hydrophilic films. Thus, it reduce the cohesiveness of the starch-protein edible film as well as decreasing the solubility of the film to water.

On the other hand, the trend showed that the solubility is directly proportional to the concentration of all three types of plasticizers. The solubility of glycerol, sorbitol and PEG_{200} plasticized film increased from 42.0039 to 67.9012%, 60.237 to 66.2179% and 42.7487 to 76.4822% when the concentration of plasticizers increased from 10 to 20% t 1:1 ratio of sago starch-gelatin

Based on Thawiem (2013), the trend of increasing in solubility with the increasing concentration of plasticizers might be due to the hydrophilic characteristics of the plasticizers that will enhance the solubility of film produced. In addition, it could be explained that at high concentration of plasticizers, more molecules of plasticizer were untrapped in the sago starch- gelatine cross linked network and able to escape into solution while at low concentration of plasticizers, there are lower untrapped of plasticizers molecule in the crosslinked network and less ability to escape to solution

While, based on the three types of plasticizers, PEG_{200} plastisized film had high solubility compared to glycerol and sorbitol plasticized film at 15, 20, and 25 % concentration of plasticizers. For instance, at 15 % concentration of plasticizers, the solubility of PEG_{200} plasticized film is 85.9336% compared to 53.9336% and 69.4298% for glycerol and sorbitol respectively. Nevertheless, at low concentration of plasticizers which is 10%, sorbitol plasticized film had higher solubility than glycerol and PEG_{200}. Thawiem (2013) stated that PEG_{200} plasticized film had high solubility because PEG_{200} had a ring and high molecular weight, which will delay the insertion between the starch-protein chains. Therefore, it will result to the ease its escape into solution.



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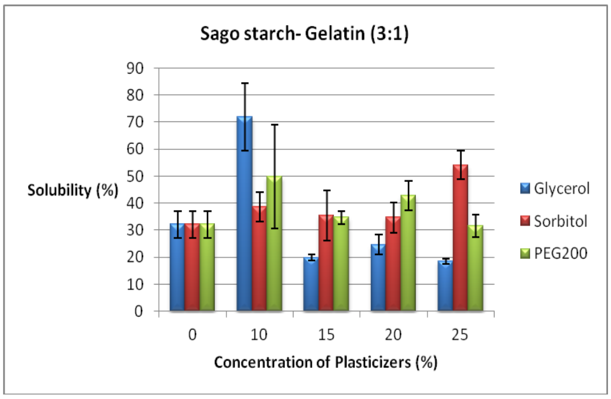

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Figure 3: Solubility of edible film of different types and concentration of plasticizers at different ratio of sago starch-gelatin

C. Water Vapor Permeability

The water vapor permeability (WVP) is the most significant property for edible films. It is required avoid or at least to decrease moisture transfer between the food and the surrounding atmosphere which also means that WVP should be as low as possible. Figure 3 showed the relationship between the concentration of plasticizers and the ratio of sago starch-gelatin to the water vapour permeability (WVP) of the film. The graph illustrated that as the content of gelatin decrease from ratio 1:1 to 2:1, the WVP decreased from 7.25×10^{-5} to 4.46×10^{-5}, 6.12×10^{-5} to 5.13×10^{-5} and 1.6×10^{-4} to 7.01×10^{-5} g.mm/ KPa. cm^2.h for 10, 15 and 20% for glycerol plasticized films.

According to Al-Hasan and Norziah (2012), this might be due to the interaction of gelatin with starch chains breaking the unbroken network of starch inter chain hydrogen bonding. Stronger attraction of water molecules expected in the films because gelatin is more hygroscopic than starch, thus resulting in higher water diffusion with higher WVP.Also, the graph showed that the WVP of the composite edible film did not show any significant differences since the reading become increase and decrease when the concentration of glycerol were increased. There was no clear trend observed in WVP for film with glycerol.

Based on Al-Hasan and Norziah (2012), the high value of WVP at high gelatin content may be due to high water diffusion resulted from high affinity of water molecule because gelatin is more hygroscopic than starch. Therefore, film with higher amount of gelatin will absorb more water molecule from the environment.

At 1:1 ratio of sago starch- gelatin, the results illustrated that WVP of PEG$_{200}$ plasticized films does not produced a constant trends since the value of WVP increased and decreased all the times. As an example, for glycerol plasticized film, as the concentration of glycerol increased from 10 to 15%, the WVP decreased from 7.25×10^{-5} to 6.12×10^{-5} while, when the concentration increased from 15 to 20%, the WVP increased from 6.12×10^{-5} to 16.00×10^{-5} g.mm/KPa. cm2.h. The similar trends occurred to sorbitol and PEG$_{200}$ plasticized film and it is contrast with Thawiem (2008)'s finding where he said that the WVP of the edible film will raise when the concentration of the plasticizers increased.

This unstable WVP trend of composite edible film proved that the addition of various type and concentration of plasticizer does not brought any effect to the WVP of the films. Nevertheless, the WVP of the composite edible film was strongly affected by the amount of gelatin added. This is because gelatin is hygroscopic material where it has ability to absorb more moisture from the environment than starch. As a result, the WVP will increase when the amount of gelatin is increase in composite edible films.

According to Bodzemir and Tutas (2003), PEG$_{200}$ is less effective in reducing intermolecular hydrogen bonding between sago starch-gelatin chains, thus, it decreases the spacing between the chains. Consequently, the WVP of PEG$_{200}$ plasticized increased when the concentration of PEG$_{200}$ increased. At the same time, the WVP of the composite edible film plasticized with PEG$_{200}$ showed reduction when the content of gelatin decreased from 1:1 to 2:1 ratio of sago starch-gelatin regardless the concentration of PEG$_{200}$. For instance, at 10 % concentration of PEG$_{200}$, the WVP of the edible film drop from 10.9×10^{-5} to 6.01×10^{-5} g.mm/KPa. cm2.h when the ratio of sago starch-gelatin transformed from 1:1 to 2:1. This showed that the reduction of gelatin content in the composite edible film increases the WVP of the film. According to research done by Al-Hassan and Norziah (2013), they suggested that gelatin absorbs more moisture from environment than starch.

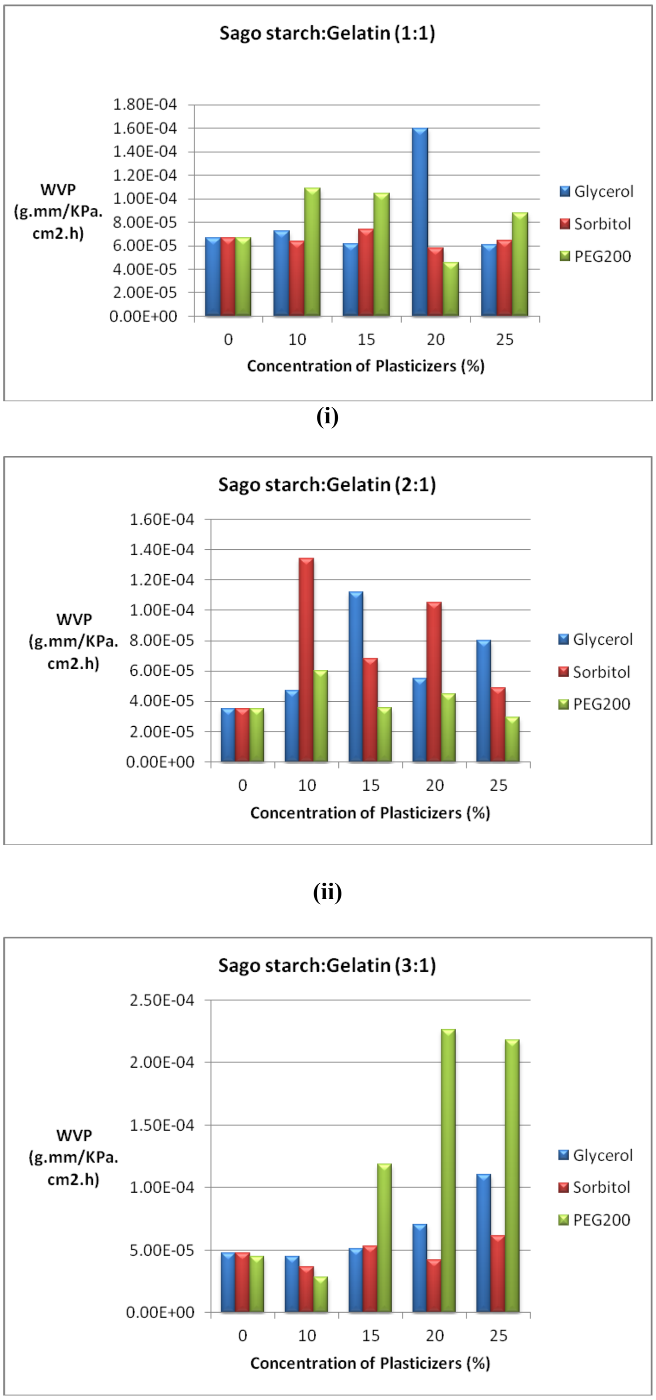

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Figure 4: Water Vapour Permeability (WVP) of edible film of different types and concentration of plasticizers at different ratio of sago starch- gelatin

I. CONCLUSION

All the objectives have been achieved in this experiment The production of composite edible film from sago starch and gelatin with addition of 0, 10, 15, 20 and 25% concentration of glycerol, sorbitol and polyethylene glycol (PEG_{200}) affect the mechanical properties, solubility and water vapour permeability (WVP) of the films. It is showed that, the addition of gelatin into the formulation increased the tensile strength of glycerol and sorbitol plasticized film, solubility and WVP of all types of plasticized films. Besides, the increasing of concentration of plasticizers decreased the tensile strength and solubility of glycerol and sorbitol plasticized films. The EAB of the edible films increased when the concentration of sorbitol and PEG_{200} increased but decreased with the concentration of glycerol. The increasing concentration of plasticizers does not produce any significant affect on the WVP of the composite edible films. It was also observed that, the edible films plasticized with glycerol has higher EAB and higher solubility when plasticized with PEG_{200} compared to others.

ACKNOWLEDGMENT

Thank you to my beloved supervisor, Madam Fariza binti Hamidon, lab technicians, Mr Irwan and Mr Ridhwan as well as Universiti Teknologi Mara.

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