Performance of Emulsified Gelatin Films for Food Packaging

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***Abstract*—Renewable sources such as lipids, polysaccharides and proteins from edible film has potential to increase shelf-life and to maintain microbial safety of food products. It also able to control moisture content, good barriers to gases and as carriers of functional ingredients such as antimicrobial and antioxidant agents. Sunflower oil (lipid-based) is used in order to form good water vapor barrier properties of edible film due to its hydrophobic nature. Other raw materials are glycerol as plasticizer and gelatin. The purposes of this study were to prepare emulsified edible films using sunflower oil as an emulsifier and to determine the properties of emulsified films such as tensile strength and elongation, thickness, moisture content, color and infrared spectroscopy. In this study, materials that will be used are gelatin, sunflower oil as emulsifier, distilled water as a solvent and glycerol as plasticizer. Results will be measured for different concentration values of sunflower oil. Properties that will be determined are tensile strength and elongation, thickness, moisture content, color and infrared spectroscopy. The objectives of this study are to prepare emulsified edible films using sunflower oil as an emulsifier to determine the properties of emulsified films such as tensile strength and elongation, thickness, moisture content, color and infrared spectroscopy.**

Keywords— gelatin, glycerol, moisture, sunflower oil

I. INTRODUCTION

Films produced with one component will no show good water barrier properties, thus composite emulsion-based edible materials need to be produced from hydrocolloids and lipids which gives better results to prevent moisture loss. If there is only mixture of lipids and hydrophilic protein or polysaccharide films, film strength can be negatively affected which proved by measurements of tensile or puncture strength.

A. Gelatin film

Gelatin is a type of protein that commonly applied in the pharmaceutical and food industry due to its low prices and large amount (Baldwin, 2012). Functional properties of gelatin are very suitable in producing edible and biodegradable films Baldwin, 2012). Nevertherless, gelatin has its own weakness where it shows high water vapor permeability. High concentration of gelatin will increase solubility of film.

Generally, the presence of gelatin in film will increase film thickness, mechanical strength, water solubility and water vapor permeability. Gelatin causes a film to become more transparent because the existence of polymeric matrix which becomes larger due to its triple helical structure. Thus, wide exposure of the structure towards light cause film becomes less opaque due to more penetration of light (Galus, 2015).

Edible film which consists of gelatin will not easily broken or damaged because gelatin will increase thickness of film due to increasing in dry mass in film. This condition will result high value of Young's modulus which cause mechanical resistance of the film becomes better.

Plasticizer must be added into film that consists of gelatin in order to improve film flexibility and water vapor barrier properties. In order to produce good thermal and mechanical stability of biopolymer, gelatin need to undergo modification process called crosslinking due to its properties of able to dissolve in aqueous solution. Crosslinking process involves glutaraldehyde (GTA) as chemical crosslinking agents because it shows high performance in term of collagenous materials stabilization (Galus, 2015).

High efficiency of crosslinking between gelatin and GTA can be obtained by using low amount of GTA in film. The correct concentration will produce high stability of film and good mechanical and thermal properties. The GTA-gelatin crosslinking shows large influence on the enthalpy change of wet gelatin films. Besides, it also does not show many differences if temperature of air-dried gelatin is denatured (Bravin, 2006).

B. Plasticizer

Glycerol shows high favorable towards water due to hydroxyl groups consists in glycerol which cause water to easily absorb into it and form hydrogen bond. This is because glycerol has low molecular weight also low density which cause water molecules to easily enter between polymer chains of glycerol to fill the free space between the molecules (Sanyang, 2016)

Glycerol is used as plasticizer to increase film flexibility but has high water vapor permeability. This is because glycerol causes film to extend longer due to decrease in tensile strength. Besides, glycerol also causes lots of changes towards properties of film tensile strength and water vapor barrier. The presence of high concentration of glycerol will increase oxygen permeability in film (Natta, 2004).

High concentration of glycerol makes the film to be very soft, transparent and easy to peel off from the petri dish. Even though small addition of glycerol is added, it cause tensile strength of film decreases due to lack of mechanical properties. This makes the film to easily elongate.

Due to high flexibility of film, it hards to be damaged if there is any tension or mechanical stress applied. Thus, film will be high quality if glycerol is present because it avoids damage during food processing handling (Natta, 2004).

In addition, high concentration of glycerol causes film to become thicker. This is because glycerol molecules will fill space in the matrix and form polymer together with edible film which produce high quality of film thickness (Arham, 2016).

Emulsion stability can be increased in term of better water vapor barrier and mechanical efficiency during the process of film drying. Plasticizer is used to improve the mechanical properties of protein or polysaccharide-based edible films (Musavi, 2007). However, the mixtures of water and plasticization can lead to increasing of gas and vapor through the edible films. The most common plasticizer used in edible film is glycerol.

The presence of plasticizer can increase water vapor permeability of the edible films. When glycerol molecules diffuse between the polymer chains, it increases water vapor diffusivity through film due to increase in the interchain space, thus encouraging the water vapor transmission.

One of the properties of glycerol is high hydrophilicity where it promotes the adsorption of water molecules can increase water vapor permeability of the film. The situation could get worse when high glycerol concentration is used because glycerol enable to open the polymer structure itself and cause the water permeability of the film increases.

Glycerol shows their hydrophilic properties that become immobilizes between carboxymethyl cellulose (CMC) chains and interact with water molecules (Hadi, 2011). Nevertheless, the presence of fatty acids causes immobilization of biopolymer chains which less interact with water molecules. A film that contains glycerol and high level of surfactants shows same mechanical properties as films contain large amount of plasticizers.

C. Sunflower Oil

Sunflower oil is a non-volatile oil comes from sunflower seeds. There are four types of sunflower oils which are high linoleic, mid-oleic, high oleic and high oleic. Firstly, high linoleic sunflower oil is not preferable compared to other types of sunflower oil due to less usage in food frying and only small volumes is produced in the North America. The ratio of fatty acids in this kind of sunflower oil is 68% linoleic acid, 21% oleic acid and 11% combined saturates. However, it needs to undergo hydrogenation to produce a stable form for frying. Some applications of high linoleic are liquid salad oil, margarine and shortening applications.

Next is mid-oleic sunflower oil where it consists of oleic acid for two-thirds of the fat content, 25% of polyunsaturated linoleic acid and 9% of saturated fat. Mid-oleic acid also known as NuSun is the largest volume of sunflower oil produced in United States and Canada. It is easy to found with affordable price. Besides, mid-oleic has good shelf-life and preferable to use in cooking due to high stability, low trans fats, good taste, excellent frying performance and 10% lower in saturated fat (Kleingartner, 2002)

High oleic sunflower oil consists of monounsaturated oleic acids around 80% of the total and the other 20% consists of saturated fats and polyunsaturated linoleic acid in equal proportions. This type of sunflower oil is produced through traditional breeding methods and important to retain good quality food products in long storage, high stability and good taste. High oleic also contains more vitamin E compared to other vegetable oils.

Lastly is high stearic sunflower oil where it produced through the same method as high oleic sunflower oil which is traditional breeding method. This new type of sunflower oil is produced to be a source of disaturated triacylglycerol alternatives and can show good properties without hydrogenation or transesterification (Rafael, 2012). One of the advantages of this oil is they act as replacement for partially hydrogenated oils or tropical oils with a higher saturate level. High stearic sunflower oil was applied in baking, margarines, ice cream and chocolate. Fatty acid contains in this oil is 18% stearic acid, 72% oleic acid, 5% linoleic acid and 5% other saturates.

Water vapor barrier properties of edible film are improved by adding sunflower oil by emulsification to. Besides, the presence of sunflower oil in a film also helps in controlling moisture loss that will retain the film structure (Maria, 2008). Oil can be modified so that it will be able to form high quality of film and not only as emulsifier. After modification, oil can be used as corrosion inhibitor and lubricating components. Example of modifiers are organic amines, salts of amines and carboxylic acids and fatty amines (*Balyts'kyi, 2013).*

Emulsion can be defined as combination of two immiscible liquids where one type of liquid being dispersed into the other liquid which is in droplet form. In addition, emulsifying agents are used in edible film in order to from a stable emulsion and efficiency of emulsified film can be increased by adding surfactants. Cosmetic emulsions use sunflower oil because there are high concentration of Vitamin E in this oil (Amar, 2007).

II. METHODOLOGY

A. Materials

Materials and chemicals that will be used in this study were sunflower oil as emulsifier, gelatin, glycerol as plasticizer and distilled water as a solvent.

B. Apparatus

Apparatus used were Universal Oven (Model UFE: 500), Hot Plate (model: Corning PC-420D), Pioneer Precision Balance and Ultrasonic Homogenizer (model: 300 V/T).

C. Preparation of samples

Firstly, 5 g of gelatin was weighed in a beaker. Then, 75 mL distilled water was added into the beaker. The conditions for stirring was at 90°C and 500 rpm. Mixing process of gelatin and distilled water is carried out for 25 minutes. After that, 10 mL of glycerol was added into the beaker and keep on stirring for 10 minutes. 0.3mL of sunflower oil was added and stirred for 25 minutes. The solution formed was homogenized using ultrasonic homogenizer for about 10 minutes using power of 10 W. 15 mL of the solution formed was transferred into petri dish. After that, the petri dishes was placed in the universal oven for drying process at 55°C for 24 hours.

D. Tensile properties

Tensile strength (TS), Young's modulus (YM) and elongation at break of the films (60mm length x 25 mm width) in size were determined using Tensile test machine (ASTM D638, ISO 527-1). The film specimens were mounted in the self-tightening roller grips of the testing machine and stretched at the rate 1mm/s until breaking. The initial distance of separation was adjusted to 50mm. The analysis was made at ambient temperature and relative humidity of (50±5) %.

E. Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) analyses were determined using a Vertex-70 spectrometer with attenuated total reflectance (ATR) accessory at 25°C. The films were placed in the support and pressed by a measuring sensor. The FTIR spectra were recorded in the wave number range of 600-4000cm^{-1} with a spectral resolution of 4cm^{-1}. The spectra obtained were used to verify the interactions between gelatin, glycerol and sunflower oil.

F. Moisture content

Moisture content (MC) of films was determined using oven which the film dried at 55°C for 24 hours. The result obtained was percentage of initial film weight lost during drying. Moisture content is calculated using the following equation:

$$\frac{W_o - W_f}{W_o} \text{ x } 100 \rightarrow \text{eqn. (1)}$$

Where W_o was the sample weight before drying while W_f was its weight after drying.

G. Color

Film surface color was measured using a Minolta chroma meter. The equipment was set up for illuminant D65 and 10°C observer angle.

Ten readings of L*, a* and b* values were measured across for each sample and average values were calculated. The total color difference was calculated using the following equation:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \rightarrow \text{eqn. (2)}$$

Where ΔL*, Δa* and Δb* are the differentials between a sample colour parameter and the colour parameter of a standard used as the film background. Each parameters has their own range values which are L (luminosity) range values are from 0 (black) to 100 (white), a values range are from -80 (greenness) to 100 (redness) and b values range are from -80 (blueness) to 70 (yellowness) (Babak, 2011).

H. Thickness

Each film thickness was measured using analogue micrometer.

III. RESULTS AND DISCUSSION

A. Infrared spectroscopy

The Fourier transform infrared spectroscopy (FTIR) was applied to examine the interactions between gelatin, glycerol and sunflower oil (SO). Figure 1 shows the FTIR spectra of the different concentration of SO in film samples. The peak at 3272.29cm^{-1} was observed due to its highest value among all the spectra values of the emulsified film. The absorption band shows group of alcohol (O-H stretching, broad) where the range is between 3550-3200cm^{-1}. The peak indicates the existence of triglyceride molecules of sunflower oil.

Band peaks between 2850-3000cm^{-1} were related to C-H stretching vibration and strong intensity. Thus, the absorption peak at 2924.37cm^{-1} can be correlated with hydrophobic interactions due to higher peak intensity of the emulsified film in Figure 1. The resulted peak is match with the trend value of moisture content because hydrophobicity of the emulsified edible film is increasing as amount of SO added higher. Thus, water vapor permeability of the films decreasing which cause moisture content can be maintained.

Band peaks between 1670-1820cm^{-1} were related to C=O stretch which has strong intensity. For example, spectrum graph of film with 0.4mL SO shows 1740.13 cm^{-1} where this peak situated in ester group. All spectrum graphs show band peaks between 1670-1820cm^{-1} because the existence of vegetable oil (SO). However, higher amount of SO added in the film do not cause higher value of the peak as shown in Figure 1. The peaks indicate how well the solution is mixed for each samples. The highest peak shows that interaction of oil and other substances in the film is well-mixed by using homogenizer (Carpine, 2016).

All samples show peak range of 1031 cm^{-1} where the results indicate the presence of O-H groups which match with the glycerol existence as a plasticizer in all films. Peak range that indicates the existence of glycerol does not have much different in values to same amount of glycerol is added in all films.

Hydroxyl group (O-H groups) peak in the spectrum shows hydrophilic behavior of glycerol and due to hydrogen bonds formed by the hydroxyl group. All films indicate band peak between 3000-3600 cm^{-1} where the peak category is O-H and N-H stretching vibrations. The peak located in this band for example for film of 0.5mL oil which is 3263.10 cm^{-1}, the peak is categorized in hydrogen bonding between protein chains and water and the interaction between protein and oil.

Each samples show different values of peak for hydroxyl group due to interruption between protein interactions which is gelatin (Ghanbarzadeh, 2011). The resulted peak also due to incorporation of oil in the film structure. This band was evidenced in moisture content part where SO take action as moisture barrier in the film.

Band peaks between 1350-1480 cm^{-1} were related to alkane which has variable intensity. All samples show peak between this range. The peak value indicates that the emulsified edible films consist of hydrocarbon which comes from the sunflower oil. In the FTIR analyses, differences in absorbance intensity between all film samples were easy to identify due to obvious peak values differences. This is because during the formation of the film, some bonds such as protein-protein bonds may break and form other bonds such as protein-oil or glycerol-oil (Jamieson, 1922). All bonds that break and formed were indicated in absorbance intensity.

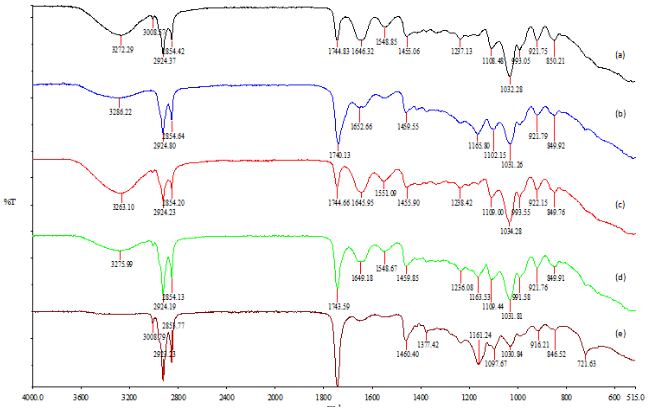

Figure 1: Infrared spectroscopy of all film where (a) 0.3ml sunflower oil (b) 0.4ml sunflower oil (c) 0.5ml sunflower oil (d) 0.6ml sunflower oil (e) 0.7mL sunflower oil

B. Colour

Colour parameters are important for first impression of the customers to buy food products. Therefore, action is taken in order to produce high quality of food film appearance where colour parameters such as L*, a* and b* were analysed and the total colour difference (ΔE) was determined due to existence of lipid in the film. Table 1 shows the values of the optical parameters for all films made by different volume of sunflower oil.

Table 1: Color parameter

Sunflower oil volume (mL)	L*	a*	b*	ΔE
0.3	34.92	0.60	-0.73	11.13
0.4	32.47	0.66	-1.85	13.63
0.5	31.43	0.51	-2.61	14.74
0.6	30.30	0.03	-2.21	15.81
0.7	29.76	0.82	-1.05	16.30

From Table 1, colour differences were identified using Comission Internationale de I'Eclairage (CIE) L*a*b* coordinates. A white plastic was measured as the standard values for calculations of ΔE where $L^*_{standard}$=46.03, $a^*_{standard}$=0.01 and $b^*_{standard}$=-0.63(Zhao, 2007).

All the films do not have much different L values and the pattern was slightly decreasing as the sunflower oil increasing. This is because sunflower oil itself shows pale yellow, thus increasing of oil made the film become less lightness and more opaque. All films show constant sign of a* and b* values where all parameter a* obtained were positive values while all parameter b* obtained are negative values.

Parameter a* and b* has inconsistent values for all samples. Meanwhile, values of total colour difference (ΔE) were increased for all films. Thus, increasing sunflower oil as emulsifier increasing value of ΔE.

The trend of ΔE values can also be observed for protein-based films which consist of vegetable oils and different hydrophobic substances. Besides, ΔE values were not too much different. Therefore, all the films show only little different of color and only slightly noticeable. Based on ΔE values in Table 1, it can be concluded that addition of higher amount of lipid compound increases color saturation towards the films.

C. Moisture content

Moisture content must be maintained in packaging films in order to maintain its freshness and quality. Water solubility affect the quality of film because easy to loss its moisture if water solubility is high. Table 2 shows moisture content of the films which has been dried for 24 hours at 55°C. The MC values of films were significantly decreased as the amount of sunflower oil increased. This happened because sunflower oil (SO) exists as hydrophobic substance.

The result shows that moisture content values were affected by amount of glycerol and sunflower oil. This is because lower amount of sunflower oil cause the moisture from the film easier to loss due to lower hydrophobicity. The existence of glycerol in the film cause high hydrophilicity which favors the adsorption of water molecules that lead to increase in water vapor permeability (WVP) of the film (Parris, 1995). Therefore, higher amount of SO is needed in order to reduce water vapor loss in the film so that moisture content can be maintained.

Moisture content in the films were determined by diffusivity and solubility of water molecules through film matrix. Low amount of oil which is 0.3 mL SO film allows the glycerol to take action in increasing WVP. This is because glycerol molecules cause increasing in interchain space between the polymer chains allow high water vapor diffusivity through the film, thus encourage the water vapor transmission (Yoshida, 2004).

Table 2: Mass of films and its moisture content

Samples (mL oil)	Before drying (g)	After drying (g)	Moisture content (%)
0.3	12.56	3.13	75.07
0.4	14.26	3.29	76.92
0.5	15.10	3.36	77.73
0.6	15.58	3.44	77.92
0.7	16.08	3.45	78.55

From table 2, the higher the amount of SO added, the higher the amount of moisture that can be preserved in the sample. Emulsified film that contain 0.7mL SO had the highest barrier property due to hydrophobicity nature of SO. Eventhough glycerol itself own a characteristic of plasticizer but glycerol not easily inhibit water vapor transmission.

Films with higher amount of oil showed good moisture barriers due to the hydrophobicity of the oil.

D. *Thickness*

Table 3: thickness of all films

Samples (mL oil)	Thickness (mm)
0.3	0.647
0.4	0.736
0.5	0.750
0.6	0.860
0.7	0.880

Physical characteristics of films are very important as customer will give first impression towards the colour, gloss and transparency. The colour of all films are influenced by film thickness. This is because sunflower oil consists of fatty acids where the fatty acids increased film opacity.

The higher the amount of fatty acids added, the higher the opacity of the film which its colour becomes high intensity. This is because the film thickness affect the color intensity. From table 3, the higher amount of SO added, the more thicker the film that increased its opacity. The result occurred due to light scattering effect of the emulsion.

Generally, thickness of a film was less than 0.3mm. Film thickness influences water vapor barrier performance. The higher the amounts of fatty acids added, the lower the ability of water vapor to permeable from the film. This is because as film thickness increased, the sunflower oil will resist the water vapor transfer across it.

Besides, water vapor permeability(WVP) also affected by size of lipid particles in the film matrix. Homogenizer is used in order to form homogeneous film so that smaller lipid particles were evenly distributed in the film matrix which lower the WVP. When chain length of fatty acids increased, WVP was decreased. This is because the film matrix became saturated and less amount of water molecules that can flow through the matrix structure.

Lower mobility of long fatty acids cause the film matrix to be firm and water vapor not easily flowing out from the film (Yoshida, 2004). Therefore, thickness of film is important because the higher the amount of SO added, the the higher the amount of fatty acids in the film matrix that will make the structure becomes firm and high hydrophobicity.

E. *Mechanical Properties*

Table 4 shows tensile tests and elongation at break of emulsified edible films for different volume of sunflower oil respectively. Tensile strength (TS) can be defined as the maximum tensile stress that a film can support while elongation at break (EAB) refers to maximum change in length of a film before it breaks. (Carpiné, 2016)

Table 4: Tensile strength and elongation at break for all films

Films (mL oil)	Average values of tensile strength (MPa)	Average values of elongation at break (%)
0.3	34.57	47.16
0.4	43.35	43.84
0.5	54.72	36.33
0.6	26.95	23.55
0.7	14.18	20.65

From figure 2, the bar chart shows the fluctuation of trend for tensile strength. The bar chart indicates the trend due to emulsified film microstructure with different amount of sunflower oil. This is because well distributed of lipid droplet will give strong tensile of film. When lipid droplet size is smaller, the overall film structure will have even lipid droplet that can increase it strength.

For film of 0.6mL and 0.7mL oil, the tensile strength is not increasing due to not well homogenized during preparation of sample. Thus the lipid droplets are not evenly distributed and large lipid globules are accumulated. Large lipid globules will fill the space between protein network which lower the films mobility and also cause the films harder to stretch. Besides, the interaction of protein-lipid in the film matrix cause the tensile strength of each film fluctuates. The graph shows how well the sunflower oil helps in improving the tensile strength based on its stretching ability.

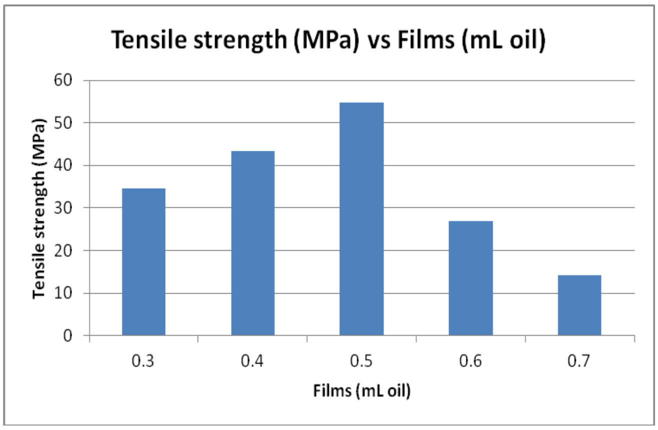

Figure 2: Bar chart of tensile strength

Mechanical testing and extensibility are important in order to maintain food quality in its packaging during storage in case existence of external stress. Lipids also can be categorized as plasticizer due to their ability to weaken the intermolecular forces between adjacent polymeric chains that will affect the results of mechanical assessment (Ghanbarzadeh, 2011). Besides, lipids can also help in improving elongation at break for edible films (Zahedi, 2010).

Table 4 also shows the trend of elongation at break values where elongation percent is decreasing as the volume of sunflower oil increases. This happened due to emulsified films is low polarity and lack of water content compared to protein films (Zahedi, 2010). The existence of glycerol as plasticizer will help in improving elasticity and flexibility of the films due to their high mobility and low molecular weight.

Water is the best plasticizer due to its polarity (Lin, 2007). For example if water interacts with proteins, it will form strong bonds thus form strong interaction that will increase values of elongation at break. In this research, glycerol is used as plasticizer thus sunflower oil will form bond protein (gelatin) that will form weak molecular structure thus decreasing percentage elongation.

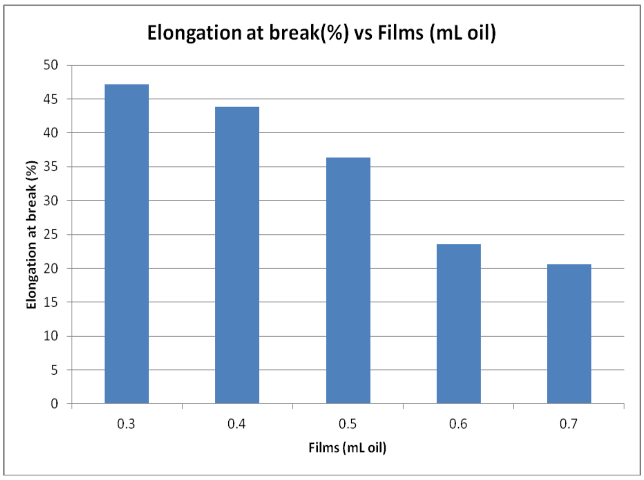

Figure 3: Bar chart of elongation at break

CONCLUSION

The characteristics of edible films are mainly affected by glycerol and sunflower oil. The existence of glycerol in film solution decreased mechanical strength and increased the water vapor permeability due to its hydrophobic nature. However, increasing concentration of sunflower oil reduced the WVP and increased the mechanical strength of the edible films. Therefore, correct amount of and proportion of glycerol, sunflower oil and gelatin plays important role in reducing water vapor transmission through the edible films.

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