



Comparative Studies of the Edible Film Based on Low Pectin Methoxyl with Glycerol and Sorbitol Plasticizers

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Abstract

This study aims to compare the characteristics of mechanical and water vapor permeability of edible film based on low pectin methoxyl from cocoa skin with glycerol and sorbitol as plasticizer. In the research also added CaCO_3 filler with the weight variation of 0; 0.2; and 0.4 gr. Pectin from cocoa peel was isolated by extraction use ammonium oxalic at a temperature of 85°C , pH of 3.6 for 60 minutes. An edible film synthesized at a temperature of 85°C to the agitation time of 50 minutes. 200 mesh of pectin used with the variation of glycerol and sorbitol plasticizer concentration are 1, 2 and 3% in volume. Edible films produced were dried at a temperature of 55°C for 6 hours. The results of the study obtained in 0.2 gr CaCO_3 concentration and 1% glycerol of edible films has a tensile strength of 0.3267 mpa, percent elongation of 12.84%, modulus young of 2.5441 mpa, and the water vapor permeability of $4.1676 \text{ g/m}^2\cdot\text{day}$. While in 0.4 gr CaCO_3 concentration and 1% sorbitol of edible films has a tensile strength of 6.511 mpa, percent elongation of 2.419%, modulus young of 269.119 mpa, and the water vapor permeability of $5.583 \text{ g/m}^2\cdot\text{day}$. Based on percent elongation characteristics, glycerol plasticizer made higher elasticity than sorbitol plasticizer. While the addition of filler able to increase tensile strength two times larger than without filler.

INTRODUCTION

The characteristics of a packaging material influenced by the edible film composition. The main composition of edible film material, are hidrocolloid, lipid and composite (Bureau, 1996). Hidrocolloid is the form of polysaccharides or proteins. One type of polysaccharide that is used in the manufacture of edible film is pectin. Pectin consists of a low and high of methoxyl pectin.

According to Olivas et al. (2009) low methoxyl pectin commonly used for the manufacture of edible coating due to the ability to form a strong gel reacts with multivalent cation such as calcium. Low methoxyl pectin can be obtained from the waste cocoa peel. Cocoa peel contains 18% pectin, 2% tannin, anthocyanin 1.04% and the rest is water (Efendy et al., 2013) so

that it can be utilized as an alternative to making edible films. Low Methoxyl Pectin (LMP) can be obtained from the peel of cocoa (*Theobroma cacao* L.) by means of extraction using ammonium oxalate (Erika, 2013). Other studies conducted by Efendy et al. (2013) about edible film using pectin from cocoa peel.

Pectin obtained by extraction using chloride acid. The results of extraction obtained are not pure pectin (protopectin) and the edible films that generated very easily soluble in water. This shows the low resistance to water, thereby affecting the power save for food that is coated with it. Based on the research of Efendy et al. (2013) shows that the edible film from pectin as a raw material has low mechanical characteristics. To improve the mechanical characteristics of the edible film can be used fillers, which serves as an amplifier. Then

Widyaningsih et al. (2012) using calcium carbonate as a filler in the composition of the edible film from the starch of banana peels. The results of this research suggest calcium carbonate as a filler is able to enhance the tensile strength, Water Vapor Permeability (WVP), and tear resistance of edible films.

Besides filler as the additional material, glycerol and sorbitol was added as plasticizer. Sudirman et al. (2012) conducts research syntheses of edible film from high methoxyl pectin from banana peel extraction with the addition of glycerol. The results showed increased elongation and decrease of the edible film tensile strength significantly. Edible films with the addition of 20% weight glycerol is recommended as the best concentration because it has the high elasticity and capable of packing of groceries.

Based on the description above, this research was conducted on the variation in the composition of the edible films to improve the mechanical characteristics. In this Study, edible film made from low methoxyl pectin from the cocoa peel with calcium carbonate as a filler, and glycerol and sorbitol as plasticizer, so that it can produce edible film in accordance with the standards of the edible film that can be applied as food packaging.

EXPERIMENTAL

Materials

The tools used in this research include: blender, grinder, sieve, thermometer, digital pH-meter, digital balance, Desiccator, molded, zip bag lock and oven. As for the materials used are cocoa peel (Desa Limau, Kabupaten Tanggamus), sodium metabisulfite 0.1%, ammonium oxalate, ethanol 96%, pH indicators, CaCO₃, aquades, glycerol and sorbitol. There are several stages performed, i.e. preparation of raw material for the extraction of pectin and producing of edible films.

Pectin Extraction from Cocoa Peel

Fresh peel of cocoa diced with a thickness of 0.5 cm soaked in sodium metabisulfite solution at a temperature of 70 °C for 15 minutes. Next Sun dried during the 3 - 4 days. Cocoa peel dry milled with a grinder and sifted until 8 mesh of size. 25 grams of dried cocoa peel dissolved in 250 ml of 2.5% ammonium oxalate at a temperature of 85 °C, pH of 3.6 for 60 minutes. Furthermore cocoa peel

slurry filters with a cloth filter and squeezed. The filtrate of pectin obtained was agglomerated by adding 750 ml of 96% ethanol with 1:8 ratio for 18 hours. Sediment of wet pectin obtained filtered, squeezed, and purified by washing three times using ethanol 96%. Wet pectin dried using the oven at a temperature of 40 °C for 6 hours, then the reduction of the size with blender and sifted up to 200 mesh of size (Erika, 2013).

Concentration analysis of Methoxyl in pectin was done with titration method and calculated using the following equation:

$$\text{Kadar metoksil (\%)} = \frac{\text{mL NaOH} \times \text{N NaOH} \times 3,1}{\text{berat sampel (g)}} \quad (1)$$

Production of Edible Film

Production process of edible film adopted from Wittaya (2013) research with different kinds of raw materials. In this research used pectin from the cocoa peel with a variation of the concentration of calcium carbonate (CaCO₃). 3 g of Pectin from cocoa peel extraction and 0 grams of CaCO₃ dissolved in 85 ml of aquades, than homogeneous with 375 rpm of agitation speed for 20 minutes. The solution that has been heated at a temperature of 85 °C for 30 minutes and when the last minute added 1%vol of glycerol and 15 ml of aquades, 40 ml solution is poured in 9 cm diameter of the mould and cooled to the room temperature. The next step of the mold is dried in an oven at a temperature of 55 °C for 6 hours. Then the edible film is released from the mold and is stored in a zip bag lock. Next step above repeated for glycerol variations above 2 and 3 %vol and variety of a sorbitol plasticizer by concentration above 1, 2, and 3 %vol, and variation of calcium carbonate filler weight 0, 0.2, and 0.4 grams. So totally obtained 18 samples of edible film. An edible film obtained are analyzed of mechanical characteristics by using Universal Testing Machine, WVP analysis by using desiccator, and functional group analysis by FTIR.

Mechanical Characterization

The mechanical properties of material in this research were determined by ultimate tensile strength, elongation at break and modulus young. The mechanical properties were tested with Universal Testing Machine based on ASTM D882-91.

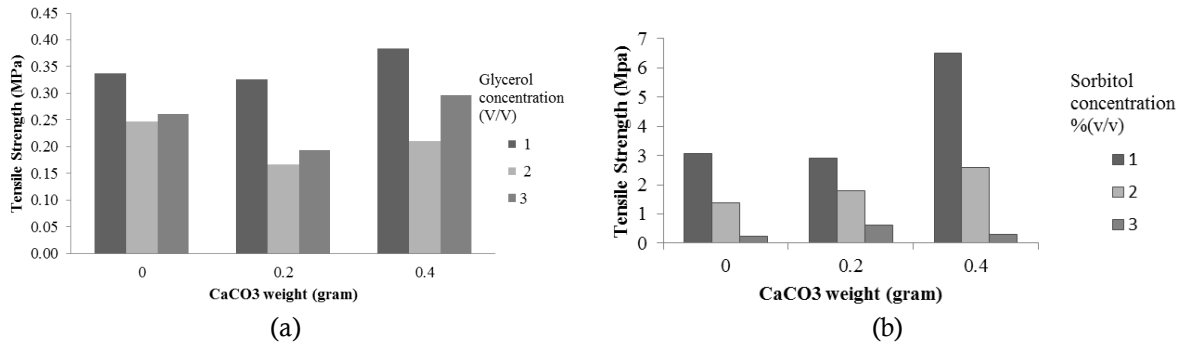


Figure 1. The influence of CaCO₃ filler weight against the tensile strength of the edible film with (A) plasticizer of glycerol and (B) plasticizer Sorbitol.

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is most commonly used on spectroscopy technique for studying the mechanism of functional group interaction that were involved in a mixture. If an organic compound illuminated to the x-rays infrared have given frequency, so the infrared frequency will be absorbed by these compounds. Many frequencies absorbed is measured as percent transmittance (%T).

Water Vapor Permeability (WVP)

This method used to refer to a method performed by Huri et al. (2014) according to ASTM E96-95, i.e edible films that will be tested should be cut and placed covered petri dish containing fruit wine. The dish that has been covered in edible film inserted into a desiccator containing silica gel that has been dried for 3 hours at a temperature of 180 °C. Measurements performed after storage for 24 hours.

Water Vapor Permeability (WVP) is calculated by the formula:

$$WVP = \frac{\Delta W}{t \times A} \quad (2)$$

where:

- WVP = Water Vapor Permeability (g/hours.m²)
- ΔW = Change the weight of edible film after 24 hours (g)
- t = Time (24 hours)
- A = Surface area of film (m²)

RESULTS AND DISCUSSION

Mechanical Characteristic

The mechanical properties test is aimed to find out the tensile strength, modulus young and the percent of elongation of edible film, so it can be known that the influence of filler plasticizer weight and CaCO₃ concentration with various kinds of plasticizer. The results of the tensile strength

analysis on the edible film product can be seen in Figure 1 (A) and 1 (B).

Based on a figure 1 (A) and 1 (B), it can be seen that the highest tensile strength value about 6.5 MPa is referred to add 0.4 gr of CaCO₃ by the sorbitol concentration of 1 %vol. The lowest of tensile strength value about 0.1661 Mpa is referred to add 0.2 gr of CaCO₃ by the glycerol concentration of 2 %vol. Based on the results obtained, The average of edible film tensile strength with sorbitol plasticizer higher than glycerol plasticizer. According to Laila in 2008, tensile strength and efficiency of a plasticizer depends on molecular weight. Tensile strength of edible film will increase with increasing molecular weight.

In terms of this, sorbitol with a molecular weight of 182.17 g/mole will have the effect of higher tensile strength of edible film than glycerol by molecular weight 92.09 g/mole. In addition, based on the functional molecule group, a group of OH- in sorbitol more than glycerol in equal concentration so intermolecular bond can increase (Wirawan et al., 2012).

Overall, the value of tensile strength of edible film decline against the concentration of plasticizer, because of the plasticizer characteristics which is able to decrease the intermolecular force along the polymer chain causing the polymer is more elastic and decrease of material tensile strength. So the addition of more than a certain amount of plasticizer will produce the film with the lower tensile strength.

In contrary, the value of edible film tensile strength increased with the additions of CaCO₃. CaCO₃ compounds as fillers on this research. CaCO₃ were added into the matrix with the aim of improving the mechanical properties of plastic through the deployment of effective pressure between fiber and matrix (Widyaningsih et al., 2012).

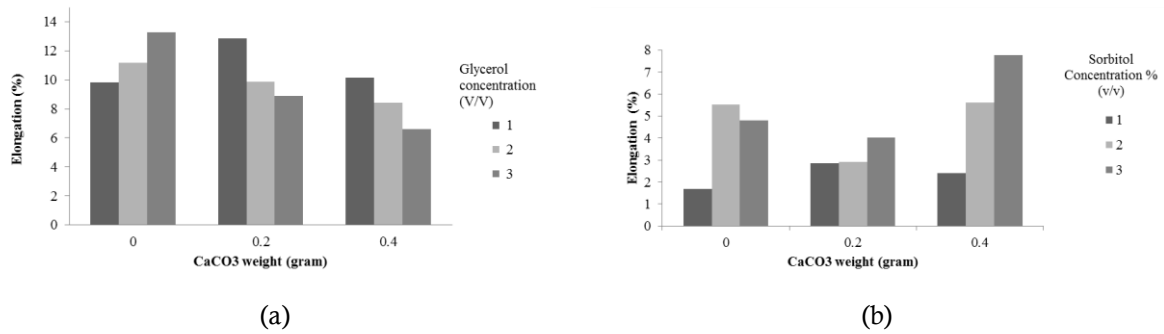


Figure 2. Decrease of filler weight CaCO₃ against percent elongation of edible film with (A) glycerol plasticizer and (B) sorbitol plasticizer

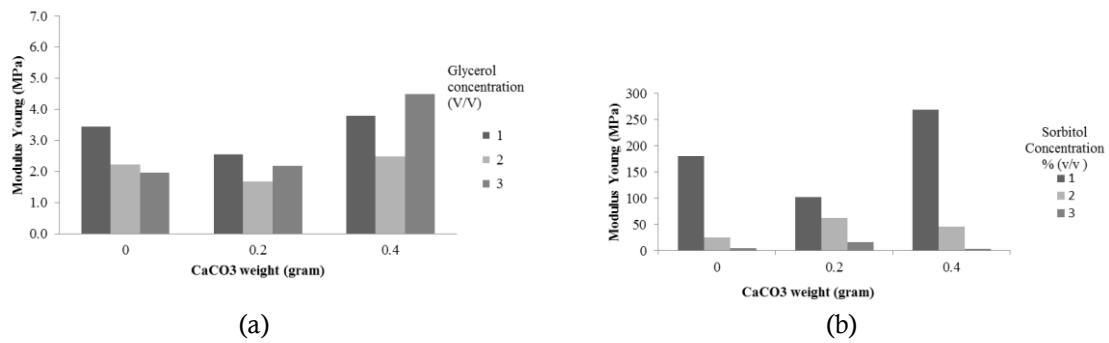


Figure 3. The influence of weight of CaCO₃ filler against the modulus young of edible films with (A) glycerol plasticizer and (B) sorbitol plasticizer

The results of the elongation analysis on edible films can be seen in Figure 2 (A) and 2 (B). Figure 2 shows that the use of plasticizer tend to increase the percentage of elongation in edible films because of a plasticizer can reduce force each of molecules and increase mobility of polymer chain (McHugh et al., 1994). In 1% glycerol concentration gives elongation higher than 2% and 3% glycerol concentration. This occurs because at concentrations of 1% mixture is saturated at a point which causes the molecules plasticizers just dispersed and interact between the structure of the starch chains causing the starch chains are more difficult to move due to steric obstruction. When the concentration of glycerol increased 2%-3% elongation decreased.

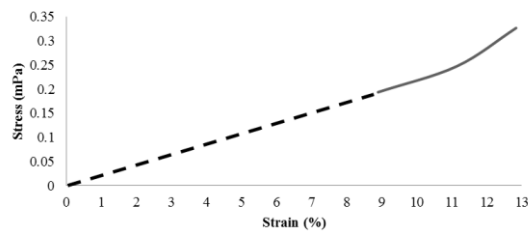
This is because the point of saturation has been exceeded, the excess plasticizers molecules reside on its own outside of the starch phase. The interaction between polymer plasticizers are influenced by the affinity of the two components, if affinity polymer plasticizers-not too strong, then it will occur plasticity between structures (distributed only among plasticizers molecule structure)

This plasticity only affects movement and mobility of structures. In case of interaction between polymer is strong enough, then the molecule plasticizers be diffused into the polymer

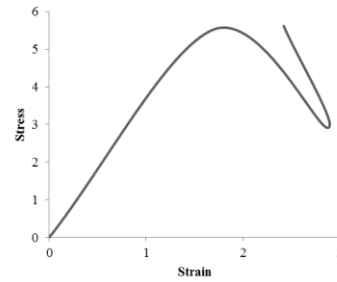
chain generates the plasticity infrastructure of intra bundle. In this case the plasticizers molecule will be among the polymer chain and affect the mobility of the chains that can increase plasticity to the limits of the compatibility of the chains that can be dispersed (dissolved) in the polymer. If the sum of these plasticizers exceeds the limits, it will happen a heterogeneous system and excessive of plasticity, so plasticity is not efficient anymore (Yusmarlela, 2009). Based on the above explanation can be said that a mixture of pectin with glycerol reaches the highest compatibility in 1% concentrations.

Based on the results of the products formed analysis, films with a glycerol plasticizer more flexible and elastic than films with a sorbitol plasticizer. 12.84% value of elongation in 1% glycerol concentration and 0.2 gr of CaCO₃ addition. This is because a sorbitol tend to form crystalline phase higher than glycerol and are more fragile. Crystallinity of sorbitol on film cause increased of tensile strength, on the other hand cause flexibility film declining (Cervera et al., 2005). However, based on the increase of CaCO₃ addition resulting in a decrease of elongation percent in edible film.

The results of the modulus young analysis of the edible film product are presented in Figure 3 (A) and 3 (B). Figure 3 shows that 1.6795 MPa is

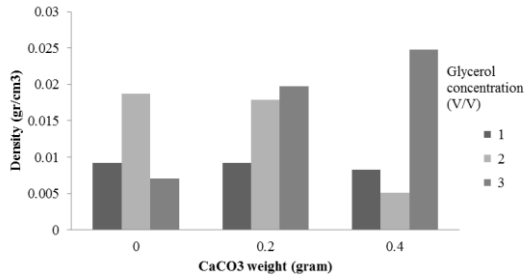


(a)

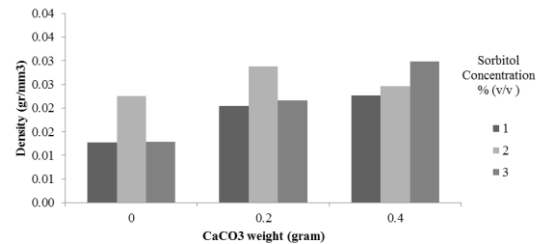


(b)

Figure 4. The curve of Stress – Strains of edible film on (A) Glycerol Plasticizer and (B) Sorbitol Plasticizer



(a)



(b)

Figure 5. Effect of the weight of CaCO₃ filler on edible films against a density with (A) Glycerol Plasticizers and (B) Sorbitol Plasticizers

the lowest modulus young when 0.2 grams CaCO₃ added by 2 % concentration of the glycerol, and the highest modulus young about 269.119 MPa when 0.4 gr CaCO₃ added by 1 % concentration of the sorbitol.

Based on figure 3 shows that the largest of filler content in an edible film can increase the stiffness and decrease elasticity. This proved that the material of a composite would be harder and powerful (Harper, 1996). Instead, with the addition of the plasticizer concentration will lead to further lower stiffness and improve the elasticity of the edible film produced. It was because of a plasticizer will form hydrogen bonds between edible film polymer bond so will interfere with the bonding between polymer and decrease stiffness distribution of fiber which is good. This led to the effective transition of the load from the matrix of the fibers (Bilbao-Sainz et al., 2011).

The uneven spread of fiber can reduce effectiveness of the amplifier (Kengkhetkit & Amornsakchai 2012). Mixing the uneven material can produce not mix matrix film, so that it can reduce the tensile strength of material (Bahri & Rashidi, 2009). The total value of modulus young in edible films with sorbitol plasticizer greater than edible films with glycerol plasticizer. Each type of polymer has different characteristics of the material.

Characteristic of polymer material can be determined by linking charts from the tensile strength and percent of elongation as a Figure 4.

In Figure 4 (A) shows that edible films with a glycerol plasticizer is the polymers that has soft and weak characteristics. A polymer that has a soft and weak character produces low modulus young, low yield stress, low ultimate strength and elongation break. While a picture 4 (B) shows that edible films with a sorbitol plasticizer is the polymers that has hard and brittle characteristics. It was because the tensile strength has increased sharply with increasing the value of elongation. However, in the maximum elongation, the tensile strength value tends to decrease. Higher of tensile strength value and lower of elongation value has indicated that the edible film having a high of modulus young. The higher of modulus young, the less capable of edible films to hold the strain of elasticity (Winding et al., 1961).

The density can in term to characterizing the physical properties of an edible film. The density can be defined as a mass per unit of the volume of materials. The density is the physical properties of a polymer. The density of an edible film in this research is presented in Figure 5.

Based on Figure 5, it can be seen that the density of edible film tends to be increased with an

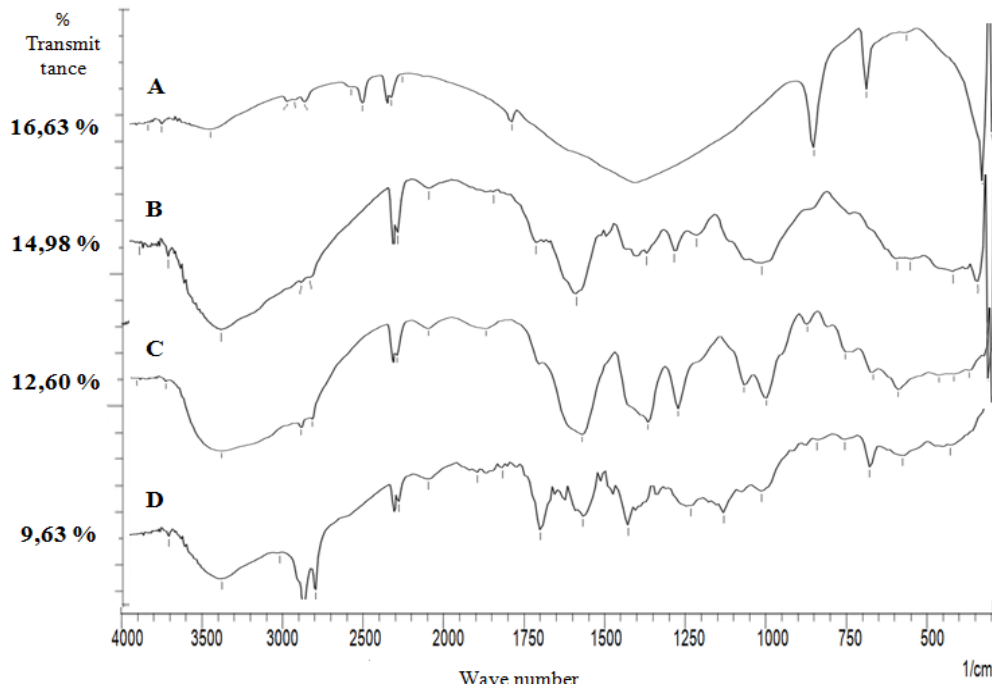


Figure 6. The results of FTIR analysis of edible film by (A) CaCO_3 filler material, (B) pectin as a raw material, (C) edible film with 1% glycerol concentration and 0.2 grams of CaCO_3 filler, and (D) edible films with 1% sorbitol concentration and 0.4 grams of CaCO_3 filler.

increase in concentration of a plasticizer. The addition of calcium carbonate increases the density of edible film, and came down from 0.2 grams to 0.4 grams. The addition 0.2 grams of calcium carbonate are considered enough to meet the empty spaces so that is increasing the density of edible film.

The best solution condition based on the result value of mechanical edible film, i.e. by 1% glycerol concentration and the addition 0.2 grams of CaCO_3 produced the density of 0.0083 g/cm^3 and 1% Sorbitol concentration and the addition 0.4 grams CaCO_3 produced the density of 0.8 g/cm^3 . The best of density will also produce the best of mechanical properties. More tied of the edible film is the more a few pore or cavity on the edible film, so that it will the higher of mechanical properties. It correlations with the result of the mechanical properties of that tends to either on the density of expected.

Functional Group Analysis of Edible Film

Based on the results of functional group analysis tests by FTIR from an edible films sample by adding CaCO_3 filler and concentration of glycerol and sorbitol plasticizer was obtained peaks that appears. Many peaks in FTIR monitor are shown that in edible films has many types of

functional groups. Figure 6, 7, and 8 has shown that the result of FTIR analysis in pectin as raw materials, CaCO_3 filler and comparison of edible film in based on the increase in concentration of glycerol plasticizer and sorbitol plasticizer by the addition of CaCO_3 .

Figure 6 shows that the FTIR analysis results from calcium carbonate, pectin and the best samples of edible film with sorbitol and glycerol plasticizer. On the results of the analysis of FTIR of edible films with glycerol, mostly there is O-H, C=O, and Ca-O functional group. Interaction of pectin, calcium carbonate and glycerol cause a vibration of the O-H functional group from 3425.58 cm^{-1} into 3410.15 cm^{-1} , the C=O functional group experienced a vibration of 1743.65 cm^{-1} into 1897.95 cm^{-1} , the C-H functional group does not change the wavelength but formed by Ca-O new bonds.

Based on the results of the analysis of FTIR samples of the best edible film with sorbitol plasticizer, most of functional group of edible films was O-H, C-H, C=O and Ca-O. Mostly interaction of pectin, sorbitol, calcium carbonate causes vibration of the hydroxyl functional group (O-H) from 3425.58 cm^{-1} into 2916.37 cm^{-1} , the vibration of the C=O functional group of 1743.65 cm^{-1} into 1735.93 cm^{-1} , the vibration of the C-H functional

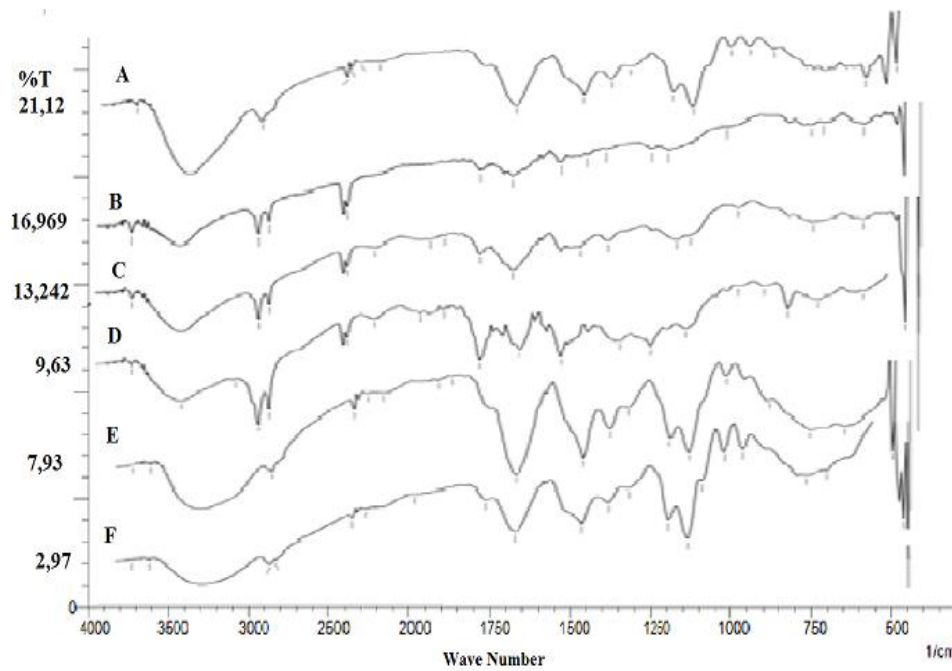


Figure 7. The FTIR analysis of edible film with various plasticizers concentrations (A) 3% of glycerol, (B) 3% of sorbitol, (C) 2% of sorbitol, (D) 1% of sorbitol, (E) 1% of glycerol, and (F) 2% of glycerol

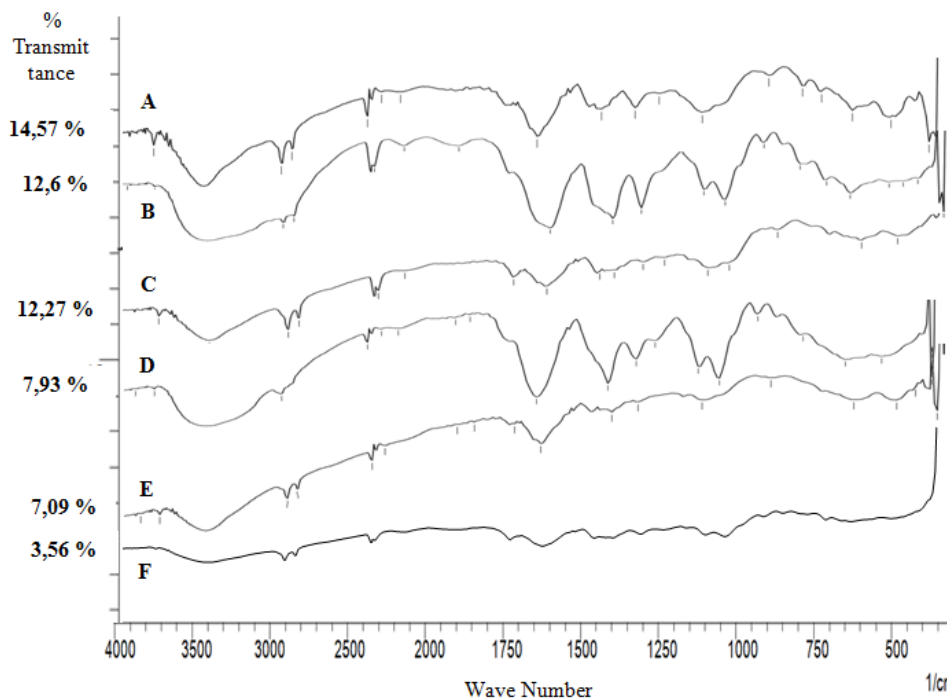


Figure 8. The FTIR analysis results of the edible films with (A) 1% sorbitol and 0.2 grams of CaCO_3 , (B) 1% glycerol and 0.2 grams of CaCO_3 , (C) 1% sorbitol and 0.4 grams of CaCO_3 , (D) 1% glycerol and 0.4 grams of CaCO_3 , (E) 1% sorbitol, and 0 grams of CaCO_3 , and (F) 1% glycerol and 0 grams of CaCO_3

group from 617.22 cm^{-1} to 624.94 cm^{-1} and formed Ca-O new bonds at a wavelength 478.35 cm^{-1} .

The results of the FTIR analysis can only be used to know the ties which is found in a compound sample. This outcome could not be used to determine the shape of the structure or quantity

a functional group of these samples. On the results of the FTIR analysis from the difference in plasticizer concentration, do not appear to be differences in a functional group as changes in the concentration of a plasticizer. Most edible films containing the functional groups O-H, C-H, C=O,

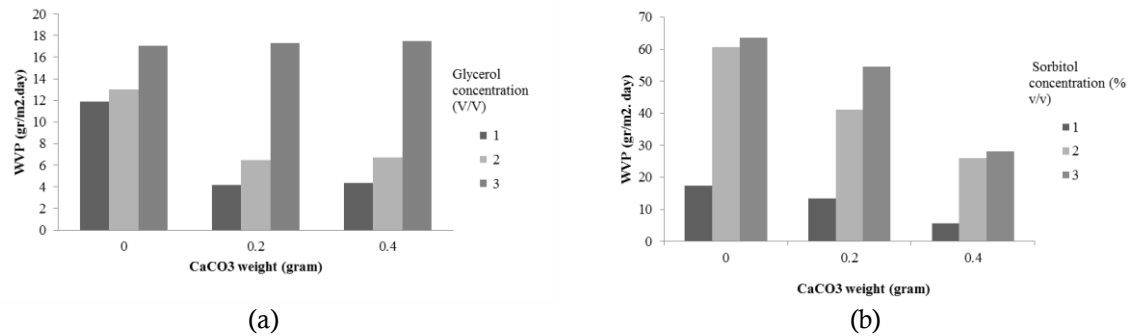


Figure 9. The influence of weight of CaCO₃ filler against water vapour permeability (WVP) of edible film with (A) glycerol plasticizer and (B) sorbitol plasticizer

and formed Ca-O as a new bond. Increase the concentration of a plasticizer followed to the decrease intensity of specific functional groups.

The functional groups O-H is changing significantly. In samples of edible films with a glycerol plasticizer, functional group O-H is at 3410.15 cm⁻¹ wavelength has vibration become 3387 cm⁻¹ as increase the concentration of glycerol. In samples of edible films with a sorbitol plasticizer, the functional group of O-H has vibration of 2916.37 cm⁻¹ become 3425.58 cm⁻¹, the functional group of C-H has vibration of 624.94 cm⁻¹ become 640.37 cm⁻¹ and 648.08 cm⁻¹, and the functional group of C-H does not have increase significant vibration as increase the concentration of sorbitol.

Figure 8 shows that the analysis results of functional group based on the type of plasticizers and calcium carbonate contained in a sample of edible film. In samples of edible film without a calcium carbonate filler, not indicated the functional group of Ca-O on the results of the FTIR analysis. Increased of calcium carbonate in samples has affect vibration on functional group of Ca-O. In samples of edible films with a glycerol plasticizer, has increased vibration of functional group from 339.47 cm⁻¹ to 354.9 cm⁻¹ as increased the addition of calcium carbonate. Vibration on functional group of Ca-O be at wavelengths 250-600 cm⁻¹ (Nasrazadani et al., 2008) other functional groups as O-H, C-H and C=O has not vibration changed as increased of calcium carbonate in samples.

The Water Vapor Permeability (WVP) Results Analysis

Water vapor permeability (WVP) is capability a film in order to curb the water vapor pass through (Wirawan et al., 2012). Methods used reference to the method by Huri et al (2014) based on ASTM E96-92. The results of the WVP analysis for edible films with the pectin of cocoa peel as a

raw material, variation of calcium carbonate filler and the concentration of glycerol and sorbitol plasticizer can be seen in Figure 9.

The results analysis of water vapor permeability as in Figure 9 shows that the higher concentration of a plasticizer will increase the value of water vapor permeability. This is because the more plasticizer content in a film will be a more open polymer structure and water molecules easier pass through the pores of the film. In addition, glycerol is hygroscopic so it magnifies the possibility of water molecules to pass through the film (Galus et al., 2012). The high permeability values found in the samples of edible films that use sorbitol as compared with samples of edible films using glycerol. It is closely related to the characteristic of the used plasticizer. Sorbitol has a greater hydrophilic properties compared to glycerol thus water molecules more easily through the sorbitol compared with glycerol.

The value of water vapor permeability tended to fall as increased filler in edible film. It is due to the content calcium will form a tissue matrix increasingly meeting so water molecules will be difficult to pass through a film (Lesmana et al., 2008). The lower value of water vapour permeability or moved closer to zero, then absorption capacity of edible film to water vapor would be smaller and better (Apriyanti et al., 2013).

CONCLUSIONS

There are several things that can be inferred from the results of this research i.e Low Methoxyl Pectin from cocoa peels can be used as an alternative of edible film raw material, the addition 0.2 gram of calcium carbonate as a filler and 1 percent glycerol concentration in edible film have met the standards of edible film based on elongation percent and water vapor permeability characteristic,

the addition of calcium carbonate as filler has improved value of tensile strength and modulus young.

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