

The Characteristics and The Making of Biopolymer Film from Oil Palm Trunk Starch (*Elaeis guineensis* Jacq.) Using Sorbitol Plasticizer

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Abstract. The research about of the biopolymers film from oil palm trunk starch had been done. Biopolymers film made by mixing of oil palm trunk starch (*Elaeis guineensis* Jacq.) and plasticizer sorbitol with the variation was 2:0,8 ; 2:1,0 ;2:1,2 (g/mL), then the film was molded on acrylic plate and dried in an oven at 40°C for 24 hours. The results of the film characterization show that the best variation of biopolymers on comparison 2:0,8 (g/mL) with the value of tensile strength was 4,26 MPa, percentage of elongation was 1,87 %, and film thickness was 0,16 mm, then it can be used as reference for addition 2 gram chitosan on the establishment of biopolymer film. It was characterized by tensile strength test with value was 14,00 MPa, percentage of elongation with value was 3,20 %, and film thickness with value was 0,10 mm. The results of Differential Scanning Calorimetry (DSC) showed increasing of the endothermic temperature was 115,75 °C and exothermic temperature was 394,56 °C. The results of Thermal Gravimetry Analysis (TGA) showed that at a temperature of 340,1 °C the film to start decomposed. The value of % Swelling was 63,176%. The Fourier Transform Infra Red (FT-IR) result showed that in biopolymers film occurs physical interaction only. As well as, the antibacterial activity analysis showed that addition of chitosan on biopolymer film made *Staphylococcus Aureus* and *Escherichia Coli* bacterial was great inhibited with index antibacterial value of 0,625 respectively. From SEM analysis showed that film had been added by chitosan more homogenous than film without chitosan addition. The presence of chitosan addition tend to increase the physical and mechanical properties of biopolymer film.

Keyword: biopolymers film, characterization, chitosan, oil palm trunk starch, sorbitol

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1 Introduction

Plastic is considered a new material as it was developed and widely used in the 20th century. The use of plastic was expanding to 220 million tons / year by 2005 (Anvenainen, 2003). This packaging material is made of petrochemical polymers, which is very popular because of its superior traits: flexible shape, transparent, not easy to break, and not corrosive. However,

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plastic polymer is not susceptible to heat and very pollutive. With the migration of its monomer components, plastic is harmful to consumer's safety and health (Coni.wanti, dkk., 2014). One attempt to reduce the use of plastic is to synthesize the degradable polymer (Darni, dkk., 2010).

The use of biopolymer such as starch is considered as an attractive solution due to its economic price, abundant availability, degradability and edibility (Mali, 2003).

Indonesia is a country with high potential in starch production. Some plants like cassava, sago, corn, sweet potato can be found in all regions of Indonesia. On the other hand, there is new innovation in starch production by using oil palm trunk. Oil palm plantation area is expanding by 11% per year, from 1.126 million hectare in 1991 to 3.584 million hectare in 2001 (Susila, 2003). Oil palm starch is a potential material to substitute the fine commercial starch, both for food and non-food (Azemi et al., 1999).

Plasticizer is the additional material in the making of film by polymer (Huri, 2013). It is defined as a non-volatile material with high boiling point, and is able to change the physical and mechanical characteristics of other materials when added to it. Plasticizer triggers the film flexibility by reducing the hydrogen bond and increasing the distance between molecules from polymer (Hui, 2006).

Glycerol and sorbitol are often used as plasticizer because they are stable and non-toxic. The addition of plasticizer can increase the flexibility and permeability towards vapour and gas (Vieira et al., 2011). However, the film-based starch has a weakness: it has weak resistance to water and moisture because of the hydrophilic nature in the starch that affects the stability and mechanism (Gracia et al., 2011). To increase the physical and functional characteristics from the starch film, biopolymer or other materials need to be added. These materials need to be hydrophobic and anti-microbial. One of hydrophobic biopolymers is chitosan, which is able to repair the film characteristics from starch, at the time to produce antimicrobial activity (Chillo et al., 2008).

Chitosan is widely used because it is biocompatible, antithrombogenic and antimicrobe. Chitosan can be printed in film as a packaging (Smith, 2005).

Solano and Gante (2014) conducted a research about the making of biodegradable film by using cornstarch with plasticizer (glycerol and sorbitol). The aim is to learn the effect of plasticizer on these two different polyols in terms of mechanical, thermal and microstructural characteristics from cornstarch biodegradable film. The result shows that plastic film with sorbitol is mechanically better in quality and contains less water compared to glycerol.

Based on the description above, the writer gains an interest to conduct a research titled "The Characteristics and The Making of Biopolymer Film from The Starch of *Elaeis guineensis* Jacq.

Using Sorbitol Plasticizer". The purpose of this study is to reduce the oil palm waste and to transform it to be eco-friendly products.

2 Materials and Methods

2.1 Equipments

In this study, the equipment used is magnetic stirrer, a set of glasses, analytical balance, hotplate, thermometer, oven, incubator, sieve, screw micrometer, vortex, autoclave, filter paper, Auto Tensile Tester, TG.A, DSC, SEM, and a set of FTIR

2.2 Materials

Materials in this research include the oil palm trunk starch, sorbitol, chitosan, aquadest, CH₃COOH 1%, nutrient, agar, microbe culture *E.coli* and *S.Aeureus*.

2.3 The Isolation of Oil Palm Trunk Starch

The pith of oil palm trunk was collected and cleansed. Then the pith was cut into pieces and mashed into powder, added with water and grinded. The mixture was then squeezed with a piece of gauze, and the starch was collected. Afterwards, the starch was left to settle as sediment before finally cleansed and filtered to ensure there is no water remaining. The starch sediment was dried in oven at 60°C temperature in 22 hours. The dried starch was grinded with pestle and mortar, then sifted using mesh sieve before it was stored to a closed container.

2.4 The Making of Acetate Acid 1%

10 mL of glacial acetate acid was measured and diluted using aquadest in a 1 Litre volumetric flask to the boundary line.

2.5 The Making of Biopolymer Film with Additional Sorbitol

2 gr of oil pal trunk starch was prepared and poured into a 250mL beaker glass, then dissolved with 50 mL aquadest and stirred at 72 °C. After the mixture was gelatinized, 0.8mL sorbitol plasticizer was added. After it was evenly mixed, the mixture was chilled and poured on the mold. Then it was dried in oven at 40 °C in 24 hours and the biopolymer *film* was taken out of the mold. The same treatment was done for sorbitol variant 1.0 and 1.2 mL. Then biopolymer film was characterized with tensile strength test and the percentage of elongation as the reference. Then the biopolymer *film* with the best variance was characterized with FT-IR, SEM, DSC and TGA, anti-bacterial and development test (% swelling) with aquadest.

2.6 The Making of Biopolymer *Film* with The Addition of Sorbitol and Chitosan

2 gr of each oil palm trunk starch and chitosan were prepared in ratio 1:1. The oil palm trunk starch was poured into a 250 mL beaker glass, and then dissolved in 50 mL aquadest, and stirred

at 72 °C till the mixture was gelatinized. Chitosan and 80 mL acetate acyd 1% was dissolved in other container, stirred for 3 hours. Then the gelatinized mixture was added to it, with additional 0.8 mL sorbitol *plasticizer*, stirred for 20 minutes. After it was evenly mixed, the mixture was poured into the mold and dried in oven at 40 °C in 48 hours. Finally, biopolymer *film* was taken out of the mold. Next, the biopolymer film was characterized using the tensile strength test parameter with the percentage of elongation, FT-IR, SEM, DSC and TGA, anti-bacterial and development test (% swelling) with aquadest.

3 Results And Discussion

3.1 The Analysis of Mechanical Characteristics

From the research on the base of oil palm trunk starch and chitosan with the addition of sorbitol plasticizer, a mechanical test was conducted in tensile test analysis. The test was conducted at room temperature, with 2000 Kgf weight and 5 mm/minute speed. The average thickness of the film is 0.1 – 0.18 mm. The tensile strength of biopolymer film was obtained as follows:

Table 3.1 The Analysis Result of The Tensile Strength and Elongation Based on Oil Palm Trunk Starch with The Addition of Sorbitol *Plasticizer*

No	Sorbitol Variance (mL)	Tensile Strength (MPa)	Elongation (%)
1	0.8	4.26	1.87
2	1.0	3.34	7.04
3	1.2	2.79	7.94

Table 3.2 The Analysis Result of Tensile Strength and Elongation of Biopolymer Based on Oil Palm Trunk Starch and Chitosan with Comparison 1:1, 2gr each with Sorbitol *Plasticizer*.

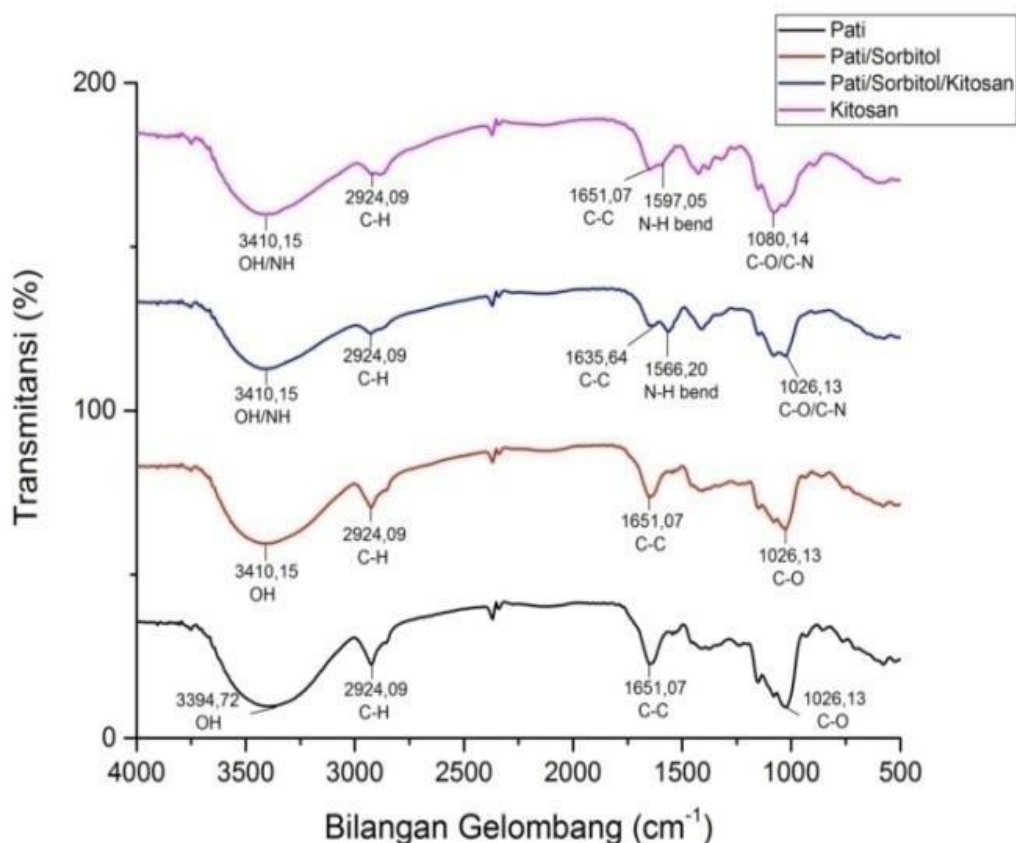
No	Sorbitol Variance (mL)	Tensile Strength (MPa)	Elongation (%)
1	0.8	14.00	3.20

Based on the tensile strength test, the highest tensile strength is seen on biopolymer film with the addition of chitosan, specifically on the starch and chitosan variation for 2 gr each and the additional of 0.8mL sorbitol plasticizer. The tensile strength is 14.00 MPa. This is because the mixture process is more stable so the *film* surface appeared to be even and stronger. Besides, chitosan as the mixing biopolymer tends to increase the value of tensile strength on certain formula. This is because chitosan is able to form hydrogen bond among the chains so the *film* becomes tighter (Setiani, 2013). This strong intermolecular bonds occurs between NH₂ from chitosan and OH from the starch. The tensile strength without sorbitol addition has higher value compared to the strength without sorbitol. *Plasticizer* is able to reduce the internal hydrogen bonds in molecules and causing the weakening intermolecular tensile strength on the adjacent polymer chains, thus reducing the breaking strength. The excessive addition of *plasticizer* will produce a film with lower tensile strength (Lai *et al*, 1997).

The elongation value determines the ability of film to stretch and elongate. According to the elongation test, the highest data is seen on the biopolymer film with 2 gr of starch and the addition of 1.2 ml sorbitol plasticizer. The value shown is 7.94%. The more sorbitol plasticizer in the polymer chains, the weaker the bonds among the polymers will be. Thus, the more flexible the film will become. (Wirawan, 2012)

3.2 The FT-IR Analysis Result on Oil Palm Trunk Starch

The FT-IR spectrum result from biopolymer film is seen in Picture 3 below:

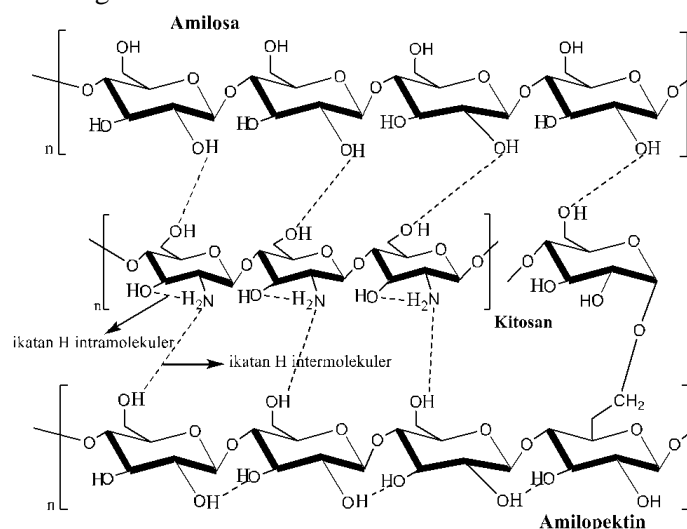


Picture 3.1 The FT-IR Analysis Result of Oil Palm Trunk Starch; Oil Palm Trunk Starch Biopolymer Starch - 0.8mL of Sorbitol ; The Oil Palm Trunk Starch Biopolymer Film – 0.8mL Sorbitol – Chitosan; and Chitosan

Picture 3.1 shows that from the starch FT-IR result, there is absorption on OH group on wavenumber of 3394.72 cm⁻¹. Chitosan FT-IR result shows absorption on NH banding group on wavenumber of 1597,06 cm⁻¹. The FT-IR result on oil palm trunk starch biopolymer film – 0.8 mL sorbitol – chitosan shows absorption on C-C group on wavenumber of 1635,64 cm⁻¹, while absorption on NH banding group on 1566,20 cm⁻¹. The absorption of OH and NH group stretching amine can be seen on the same area on wavenumber 3410,15 cm⁻¹ with some expansion., the absorption of OH group is stronger because of those sorbitol that contain plenty of OH group, causing the interaction between starch and sorbitol molecules (Setiani, 2013). The absorption by NH bonds is less intensive than the absorption by

OH, partly because the hydrogen bonds inside amine is weaker and another part is because NK bonds. are less polar. (Fessenden, 1982). The FT-IR result of oil palm trunk starch biopolymer film – 0.8 mL sorbitol shows the absorption of CH group similar to the other FT-IR results, which is on wavenumber 2924,09 cm^{-1} .

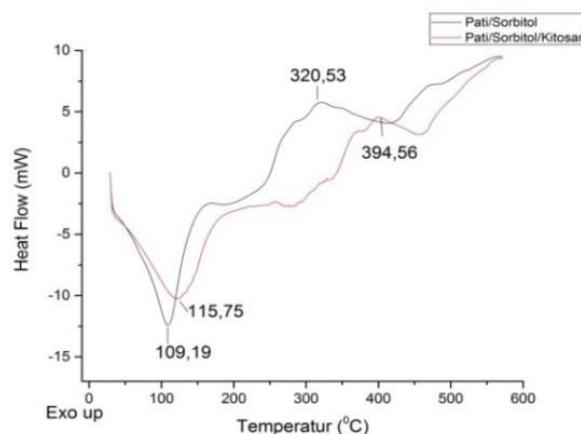
The FT-IR results show the making process of biopolymer film is a mixing process in physics with the interaction of hydrogen among chains, causing the bonding of hydrogen when atom O and atom N in chitosan interact with atom H from the amylose, amylopectin or chitosan itself. This hydrogen interaction can also happen between amyloses or between amylose and amylopectin as stated by Setiani (2013). On the other hand, the interaction between plasticizer and starch happens because the hydrogen bonds from the compatible hydroxyl functional group between the *plasticizer* and polymer (Yang *et al.*, 2000). This idea about hydrogen interaction can be seen in this following Picture 3.2:



Picture 3.2 Hydrogen Interaction Between Amylose, Amylopectin and Chitosan in Biopolymer Film (Setiani, 2013)

3.3 The Analysis Result of Differential Scanning Calorimetry (DSC) of Oil Palm Trunk Starch Biopolymer Film

The aim of DSC analysis is to study the endothermic and exothermic reactions on temperature. The DSC analysis result from oil palm trunk starch biopolymer *film* can be seen in picture 3.3 and table 3.3 below:



Picture 3.3 The DSC Analysis Chart of Oil Palm Trunk Starch Biopolymer *Film*Table 3.3 The DSC analysis of Oil Palm Trunk Starch Biopolymer *Film*

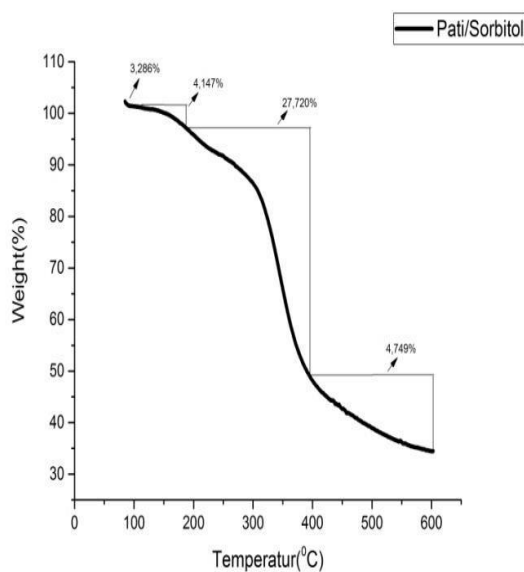
Sample	Parameter	
	Endothermic ($^{\circ}\text{C}$)	Exothermic ($^{\circ}\text{C}$)
Oil Palm Trunk Starch Film – Sorbitol 2 : 0.8	109.19	320.53
Oil Palm Trunk Starch Film – Sorbitol – Chitosan 2:0, 8:2	115.75	394.56

According to the data above, there is an increase in both endothermic and exothermic temperature after the addition of 2gr chitosan. Endothermic temperature was on 115.75 $^{\circ}\text{C}$ and exothermic on 394.56 $^{\circ}\text{C}$. It is assumed when the film forming molecular bonds get stronger with the addition of chitosan, the endothermic and exothermic temperature will arise because the temperature degradation of chitosan molecules happen above 250 $^{\circ}\text{C}$ (Martinez – Camacho, 2010).

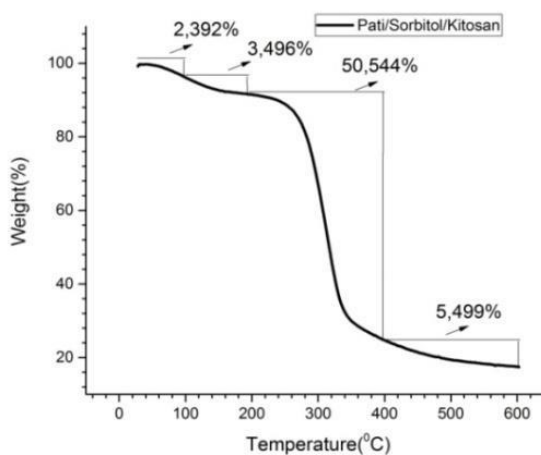
3.4 The Analysis Result of Thermal Gravimetry Analysis (TGA) of Oil Palm Trunk Starch Biopolymer *Film*

From the TGA analysis of the two samples, there is a result for oil palm trunk starch biopolymer film – sorbitol 0.8 mL. It is seen that at 100 $^{\circ}\text{C}$ temperature, biopolymer film loses its mass. It is shown in picture 3.4 that mass loss occurs in four stages. First stage is at 26.6 $^{\circ}\text{C}$ – 99.8 $^{\circ}\text{C}$, there is 3.0864% mass loss. On this stage, the volatile ingredient such as water starts evaporating. Second stage is at 100.1 $^{\circ}\text{C}$ – 199.9 $^{\circ}\text{C}$ where mass loss is 4.147%. On this stage, all water has evaporated. Third stage is the climax degradation stage at 243 $^{\circ}\text{C}$ – 329.1 $^{\circ}\text{C}$, the mass loss is 27.720%. On this stage, there is depolymeration of oil palm trunk starch and sorbitol molecules which are decomposed to CO_2 and H_2O (Anugrah 2015). The last stage is at 400.1 $^{\circ}\text{C}$ - 602.9 $^{\circ}\text{C}$, which is the last degradation process and produces 4.749% residue. While for the oil palm trunk starch biopolymer *film* – 0.8 mL sorbitol, with the addition of 2 gr chitosan, the result is relatively similar on each stage. However, on the third stage at 258.4 $^{\circ}\text{C}$ – 340.1 $^{\circ}\text{C}$, the mass loss increases to 50.544%. It is assumed that depolymeration occurred the oil palm trunk starch molecules, sorbitol molecules and chitosan, which are decomposed to CO_2 , NO_2 , and H_2O (Anugrah, 2015).

The TGA analysis result for biopolymer film can be seen at Picture 3.4; 3.5 and table 3.4 below:



Picture 3.4 The TGA Analysis result of Oil Palm Trunk Starch – 0.8mL sorbitol



Picture 3.5 The TGA Analysis result of Oil Palm Trunk Starch – 0.8mL Sorbitol – Chitosan

Table 1 Table 3.4 The TGA Analysis result of Oil Palm Trunk Starch Biopolymer *Film*

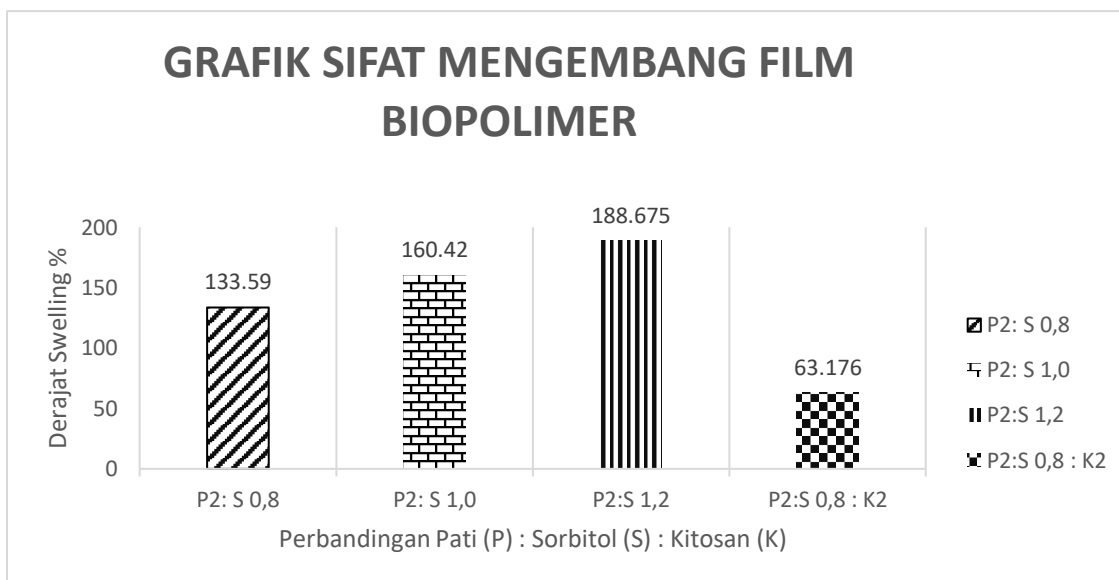
Sample	Parameter	
	Climax Temperature in Degradation (°C)	Residue (%)
Oil Palm Trunk Starch Film – Sorbitol 2 : 0.8	329.1	4.749
Oil Palm Trunk Starch Film – Sorbitol – Chitosan 2:0, 8:2	340.1	5.499

3.5 % Swelling of Oil Palm Trunk Starch Biopolymer Film

The % swelling was done by comparing the result of degree measurement between the oil palm trunk starch biopolymer film with sorbitol plasticizer and the obtained biopolymer film – chitosan. Result can be calculated by this following equation 3.1:

$$\% \text{ Swelling} = \frac{W_{final} - W_{initial}}{W_{initial}} \times 100 \%$$

The measurement result biopolymer film expansion in water can be seen in picture 3.6:

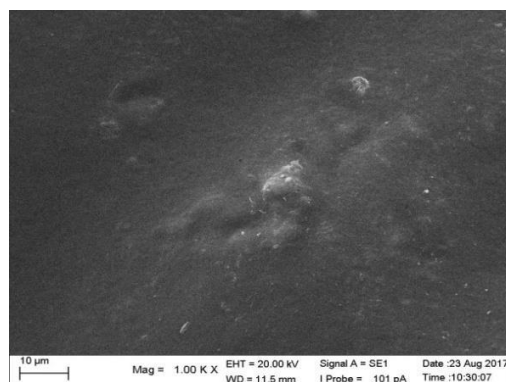


Picture 3.6 The Expansion Chart of Oil Palm Trunk Starch

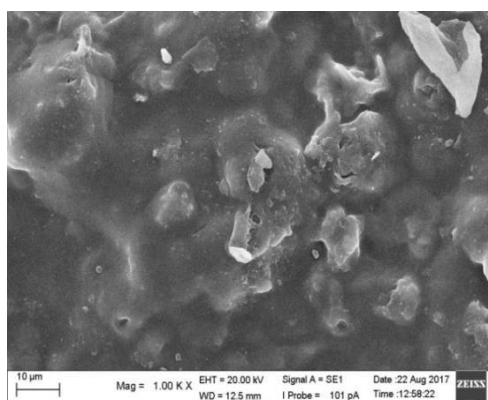
In Picture 3.6, it is seen that there is a the best mixture variance between oil palm trunk starch and sorbitol with the best resistance to water is the starch with 0.8mL sorbitol, which has the lowest expansion rate at 133.59%. Then the addition of chitosan causes the decrease in the % Swelling of the biopolymer film, reaching 63.176%. This is because chitosan is not hydrophobic and not soluble in water. The more concentration of chitosan causes the less value of % Swelling but the stronger resistance to water (Saputro, 2017).

3.6 The Analysis Result of SEM (Scanning Electron Microscopy)

The result of SEM shows the surface of biopolymer *film*, the result is influenced by the composing materials that are evenly mixed, the additional filler or plasticizer based on the highest mechanical test, then SEM analysis. The biopolymer result from the 2gr variation of oil palm trunk starch, 2 gr of chitosan with the addition of 0.8mL sorbitol and the variation of 2gr starch and 0.8 mL sorbitol.



Picture 3.7 SEM analysis on biopolymer film from oil palm trunk starch – chitosan with the addition of sorbitol *plasticizer*



Picture 3.8 SEM analysis on the making of Biopolymer film by Mag 1.00 KX.

From the SEM analysis, it can be seen that biopolymer *film* on variation of 0.8 mL sorbitol and 2gr starch shows the morphology of biopolymer film surface where it has many cavities and not perfectly mixed, but with the addition of chitosan, the significant change can be seen where cavities reduce in number and size. This is because chitosan is able to form the hydrogen bonds among chains so the *film* is tightened (Setiani, 2013). The result of SEM also shows the rough and porous surface. The rough surface indicates the homogenic film caused by the chitosan molecules triggering the stronger interaction of intramolecular hydrogens than intermolecular interaction (Xu, 2004).

3.7 The Result of Antimicrobial Activity Test on The Oil Palm Trunk Starch and Sorbitol with The Addition of Sorbitol *Plasticizer*

The antibacterial activity test was conducted on biopolymer film by using disc method, and antimicrobial index data is obtained as follows:

Table 3.5 The Observation on Antimicrobial Activity

Sample	Antimicrobial Index	
	<i>E.coli</i>	<i>S.Aureus</i>
Oil Palm Trunk Starch film – sorbitol 2 gr: 0.8mL	0.139	-

Oil Palm Trunk Starch film – sorbitol 2 gr:1.0 mL	0.257	0.105
Oil Palm Trunk Starch film – sorbitol 2 gr: 1.2 mL	0.515	0.388
Oil Palm Trunk Starch film – chitosan - sorbitol 2 gr: 2 gr: 0.8 mL	0.625	0.625

The antibacterial activity test of biopolymer *film* on table 3.5 towards *Staphylococcus Aureus* and *Echerichia Coli* shows positive result. It is marked by the clear solution around the *film*.

The test result of the antimicrobial activity shows that biopolymer film with the addition of chitosan has the biggest antimicrobial index towards *Staphylococcus Aureus* and *Escherichia Coli* bacteria because chitosan contains the reactive functional amina group (-NH₂) with positive charge, thus is able to bond with the bacterial cell wall with negative charge. On the other hand, chitosan has a structure similar to peptidoglycan, the forming structure of 90% cell wall in positif gram bacteria (Hafdani, 2011).

4 Conclusion

The characteristics of biopolymer *film* of oil palm trunk starch and sorbitol plasticizer with sorbitol variance of 0.8; 1.0; 1.2mL, tensile strength obtained are 4.26; 3.34; 2.79MPa. While the oil palm trunk starch biopolymer *film* – chitosan with the addition of 0.8 mL sorbitol plasticizer, tensile strength is 14.00 MPa. From the result of FT-IR, it can be seen that there is no chemical reaction on biopolymer *film*, only the interaction between hydrogen bonds of starch and chitosan. From DSC and TGA analysis result, it is seen that the thermal resistance of biopolymer film increases, causing 50.544% mass loss, then % Swelling of 133.59%; 160.42% and 188.675%. The addition of chitosan causing the water resistance of biopolymer *film* increases with 63.176 % Swelling. The SEM analysis result is not mixed homogenously. While from the antimicrobial analysis of biopolymer film, it can be seen that chitosan has the most antibacterial activities towards *E.Coli* and *S.Aureus* bacteria, reaching 0.625.

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