

Thermoplastic Starch Film Made from Cellulose to Extend the Shelf Life of Red Chilies

Vonny Indah Sari¹, Anania Rahmah^{2,✉}, Vivin Jenika Putri², Hanifah Ulfa Azzahro², Lukman Hakim Nasution³

¹ Department of Agrotechnology, Faculty of Agricultural, Universitas Lancang Kuning, Pekanbaru, INDONESIA.

² Department of Agricultural Technology, Faculty of Agricultural, Universitas Lancang Kuning, Pekanbaru, INDONESIA

³ Badan Riset Inovasi Nasional Lampung, INDONESIA.

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Corresponding Author:

✉ ananiarahmah@unilak.ac.id
(Anania Rahmah)

ABSTRACT

Thermoplastic starch (TPS) is a biofilm made from starch and cellulose. The advantage of thermoplastic starch is easily degraded. TPS has weaknesses in high water affinity and low mechanical properties, so it is necessary to add polyvinyl alcohol (PVA) in making TPS packaging. The mechanical properties of TPS-PVA can be further improved by modifying it into a composite with coconut fiber cellulose. This research was purposed to produces a film containing cellulose from coconut fiber as a reinforcing material for film composites as well as to evaluate the biodegradation properties of the resulting film. It is known that the addition of 5% w/v coconut cellulose fiber is the formulation with the best yield when compared with the 1% and 3% w/v formulations. Applying TPS film to chilies can increase the shelf life of chilies. As a product that spoils quickly, one of the efforts made to maintain the quality of chilies is to use packaging made from TPS. Storing chilies at different temperature conditions using TPS-PVA film packaging with the addition of 5% w/v coconut fiber cellulose was able to maintain the shelf life and quality of chilies for 19 days at cold temperatures.

1. INTRODUCTION

Currently, packaging has an important role in maintaining the quality of the product it is packaged in, however packaging made from plastic in large quantities will cause various environmental problems, because plastic made from petroleum takes a long time to decompose (Ren, 2003). This will cause land and sea pollution and poisoning of aquatic biota. Many technologies have been discovered to minimize the impact of plastic pollution, but the number of humans will continue to increase. This causes the population to increase in demand for plastic, increasing plastic production and plastic waste (EEA, 2013). So plastic components are needed with natural raw materials that are easily broken down by soil microorganisms.

Eco-product based packaging is much sought after because the level of structural complexity of some biopolymers is no different from synthetic polymers, one of which is thermoplastic starch (Wunderlich, 2011). The structure of thermoplastic starch has similarities with synthetic thermoplastic, such as being able to undergo deformation, softening when heated above the melting temperature, vitrification and changes in brittleness (Stepto, 2000). The quality and characteristics of thermoplastic starch are influenced by the chemical components amylose and amylopectin. According to Krogars *et al.* (2013), high amylose content gives thermoplastic starch properties that are more flexible, impermeable to oxygen, resistant to oil, heat resistant and soluble in water.

Plastic made from natural raw materials such as starch can be used, because plastic can be easily broken down by soil microorganisms. Starch is a biopolymer that occurs in nature and can be found in corn, cassava, sago and industrial by-products. Starch can be formed to resemble a thermoplastic material by heating and stirring at high speed. The starch gelatinization process will occur with the addition of an appropriate plasticizer (Lendvai *et al.*, 2017). The thermoplastic produced from starch is called thermoplastic starch (TPS) which has properties similar to conventional thermoplastic polymers (Stepito, 2006). Thermoplastic Starch has disadvantages such as high water affinity and low mechanical properties (Bocz *et al.*, 2014), therefore it needs to be modified by adding cellulose composites which can increase barrier, mechanical and thermal resistance properties such as silica nanocomposite (Salgado *et al.*, 2017). Cellulose can be isolated from natural lignocellulose sources, one of which is found in coconut fiber which is included in the abundant waste category. Cellulose has a straight chain polymer molecular structure of β -1,4-glucopyranose. Cellulose isolation can be done using mechanical treatment. Cellulose has the ability to reduce the coefficient of thermal expansion, increase strength, high temperature and chemical resistance (Yano *et al.*, 2005). Cellulose can be isolated from natural lignocellulose sources, one of which is found in coconut fiber which is included in the abundant waste category. Cellulose has a straight chain polymer molecular structure of β -1,4-glucopyranose. Cellulose isolation can be done using mechanical treatment. Cellulose has the ability to reduce the coefficient of thermal expansion, increase strength, high temperature and chemical resistance (Yano *et al.*, 2005).

This research aims to obtain biofilms made from coconut coir cellulose fiber with the best formulation to maintain the quality of horticultural products in cold conditions and room temperature and in the end to identify the freshness of chilies using packaging made from thermoplastic starch containing cellulose from coconut fiber as a strengthening agent for the composite film on the biodegradation properties of the resulting film. This research can improve the function of packaging which not only protects but can also maintain the freshness of chilies, extend shelf life making it easier for producers and consumers.

2. MATERIALS AND METHODS

2.1. Materials and tools

The tools used in this research included analytical scales (OHAUS PA214), chemical glasses, vials, beaker glasses, volume pipettes, ovens, magnetic stirrers, desiccators, scissors, glass plates and other analytical tools. The materials used in this research included coconut fiber, red chili obtained from the experimental field of the Faculty of Agriculture, Lancang Kuning University, distilled water, NaOH, H₂O₂, and PVA.

2.2. Design of Experiment

This research used a Completely Randomized Design with 3 replications to study the performance of thermoplastic starch composite starch and coconut fiber cellulose films. There are two factors studied, namely the length of storage time (α) in the form of the 1st (α_1), 3th (α_2), 5th (α_3), 7th (α_4) and 9th (α_5) days as well as the conditions storage (β) in the form of cold temperature (β_1) and room temperature (β_2). The statistical model used is as follows.

$$Y(ij)_k = \mu + \alpha(i) + \beta(j) + (\alpha\beta)(ij) + \rho_k + \varepsilon(ij)_k \quad (1)$$

where $Y(ij)_k$ is observation value at the i^{th} level α factor, j^{th} level β factor, and k^{th} group, μ is general mean value, $\alpha(i)$ is influence of type of packaging material at level i ($i = 2, 4, 6, 8$, and 10 days), $\beta(j)$ is influence of storage condition factors at the j^{th} level ($j = \text{cold temperature and room temperature}$), $(\alpha\beta)(ij)$ is interaction effect between factor α at the i^{th} level and factor β at the j^{th} level, ρ_k is additive group effect and is assumed not to interact with treatment, $\varepsilon(ij)_k$ is influence of errors from the i^{th} factor α , factor β j^{th} level and k^{th} group. The data were then analyzed using ANOVA at $\alpha = 0.05$ and the further test using Duncan Multi Range Test.

2.3. Coconut Coir Cellulose Isolation (Modification Tasaso (2015))

Dried coconut coir was dish-milled to reduce in size to 1 – 0.5 cm, then boiled at 100 °C for 60 min and dried using an oven at 70 °C for 12 h. Then delignify the coconut coir with 10% NaOH (1:20, w/v) at 100 °C for 60 min. The black solution was filtered and the solid residue was washed using distilled water until it was neutral. The residue was dried

in an oven at 70 °C for 12 h and a dry solid residue was obtained. Third, bleaching the delignified coconut coir using 30% H₂O₂ solution (1:10, w/v) and cooked in a water bath at 85 – 90 °C for 4 h. The bleaching results were then filtered and washed using distilled water until neutral and cellulose was obtained.

The method used to prepare starch-PVA composite films was modified from (Panaiteanu *et al.*, 2015). TPS-PVA films from coconut fiber cellulose was made using the solution casting method based on the method of Panaiteanu *et al.* (2015). The film was made based on previous research that had been carried out. Mixing the ingredients was carried out with a ratio of starch and PVA (4:1), then added 25% w/w glycerol, formaldehyde solution (cross linking agent) (3% w/w), and coconut fiber cellulose (3% w/w).

Formulation of mixed ingredients based on the weight of dry ingredients. Mixing starch, PVA, and reagents was carried out by stirring using a magnetic stirrer at a stirring speed of 1000 rpm for 20 min. The mixture is heated and stirred to 90 °C, heating and stirring is continued by maintaining a temperature of 90-95 °C for 20 min. The process continues by removing bubbles in the film solution by stirring for ± 5 min. The next process is carried out by printing on a Teflon mold and dried at a temperature of 50 °C for 24 h. This process was carried out in 3 repetitions with 3 different starch groups. The resulted bioplastic material was tested for water vapor transmission rate, water content, density, tensile strength, and solubility.

2.4. FTIR and SEM

Characteristics aims to determine the physical properties and surface condition of the pore surface of thermoplastic starch film made from coconut fiber cellulose. To determine the chemical structure of the thermoplastic starch film made from cellulose, it was observed using FTIR. Data was obtained based on variations in the concentration of added coconut fiber cellulose to the film. FTIR analysis is carried out by observing the graphs produced from FTIR analysis. The FTIR method used was to prepare a dry sample of 2 mg. The sample is placed in the holder and places it in the path of the FTIR beam and observes the graph that is formed (Putri, 2019). Cellulose fiber samples were analyzed for their morphology using Scanning Electron Microscopy (SEM) with 1000X magnification.

3. RESULTS AND DISCUSSION

3.1. Chemical Components

The chemical components of coconut coir mostly consist of hemicellulose, cellulose and lignin. Determination of the content of chemical components of cellulose, hemicellulose and lignin using the TAPPI method. The lignin content of coconut coir was carried out by a delignification process using 10% NaOH resulting in a lignin content of 21.51% (Wening, 2017). Factors that affect the acceleration of lignin degradation are temperature, pressure and concentration. Lignin degradation occurs due to hydroxy ions (OH⁻) from NaOH which will break the bonds in the basic structure of lignin, while sodium ions (Na⁺) will bind to lignin to form phenolic sodium which is polar and easily soluble (Fengel & Wegener, 1995). The lignin content which is still quite high causes the delignified coconut coir to have a dark color and a rigid structure, as shown in Figure 1.



Figure 1. Visual appearance of material: (a) coconut coir, (b) delignified coconut coir, and (c) coconut coir cellulose

Increasing the degree of whiteness without reducing its properties is done by bleaching using H_2O_2 . The color of the fiber that is still left behind as a result of delignification is generally a residue of lignin, pigment or other substances (Wening, 2017). The degree of whiteness is strongly influenced by the number of chromophores, namely functional groups. Isolation of cellulose using 30% H_2O_2 has the ability to increase the cellulose content to 90.22% (Santhi *et al.*, 2020). The increased cellulose content was caused by a decrease in lignin content and an increase in whiteness, resulting in high purity cellulose.

3.2. Characteristics of Thermoplastic Films

3.2.1. Water Vapor Transmission Rate (WVTR)

Water vapor transmission rate (WVTR) is the ability of the film to restrain the rate of water vapor that penetrates it. Film permeability is affected by concentration differences, the greater the concentration difference, the faster mass transfer occurs. It is necessary to know the permeability value of a type of film because it can be used to estimate the shelf life of packaged products and determine which products or materials are suitable for the packaging. WVTR can be determined using the slope value of the plot of the amount of water vapor lost each time divided by the area of the film (Krochta, 1997).

The WVTR value is affected by the density, the higher the density, the smaller the WVTR value. The film has a low density and an open structure, easily penetrated by fluids. WVTR coconut coir cellulose film for different treatments (control, 1%, 3%, and 5%) was 3.7862, 3.7089, 3.7532, and 3.6834 $\text{g.m}^2.\text{h}^{-1}$, respectively. This shows that the addition of 5% cellulose in film making is better than the other treatments because the test value looks higher than the other formulations. This shows that the water vapor to penetrate the film is getting less at room temperature. The difficulty of penetrating water vapor is caused by the strong bond between the film constituent materials. Equistar (2003) states that the permeability of packaging can be affected by the crystalline structure of the plastic. At low temperature conditions, the bond structure of the crystalline is strengthened so that it is more resistant to water vapor permeability.

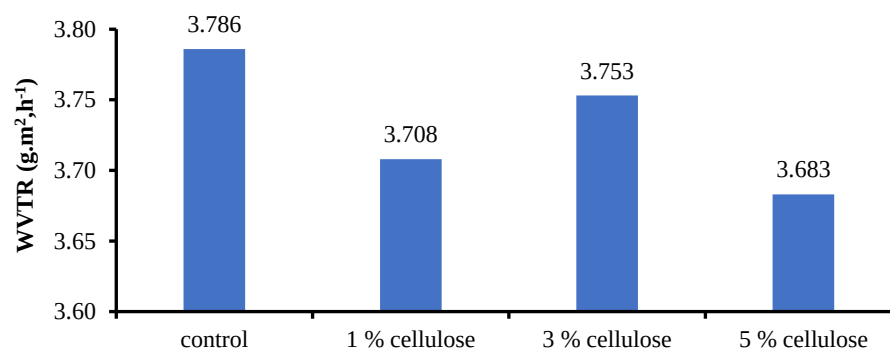


Figure 2. Graph of the effect of adding cellulose to the manufacture of thermoplastic films

The improvement in the WVTR properties of the TPS film was due to the use of cellulose as a filler material. Cellulose causes the direct diffusion path of water vapor into the composite to become tortuous (tortuous path) thereby blocking water vapor from passing through the film (Lani *et al.*, 2014). The best WVTR test result is with 3% formulation. Based on the results of the variance value of 0.576, it indicated that the addition of coconut coir cellulose to the film had no significant effect.

3.2.2. Tensile Strength

The ability of a film to accept a load or force and then be able to maintain it before breaking or without damage can be measured by tensile strength. According to van Vlack (1991), tensile strength is affected by the reaction of polymer bonds with atoms or secondary bonds with polymer chains against external forces. The tensile strength value of thermoplastic films made from cellulose from coconut coir through the addition of cellulose can be seen in Figure 3.

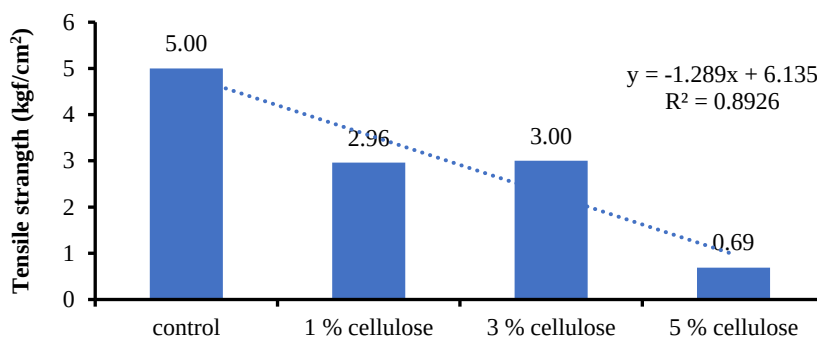


Figure 3. Graph of the effect of adding coconut coir cellulose to the tensile strength of the film

The results of the tensile strength test of variance of the film with an R^2 value of 0.891. The results of the ANOVA test showed that the addition of cellulose had a significant effect on the tensile strength of biofilm with a p value of 0.001. A good film has a tensile strength value with maximum length, this determines the elastic properties of the resulting film. The longer or higher the value resulting from the elongation measurement, the higher the elasticity or flexibility value of the film.

3.3. Solubility

The results of the water absorption test showed that there was an effect of the addition of cellulose on the characteristics of the thermoplastic film. This is confirmed in Figure 6 which shows that there is an increase in the 1% dan 3% treatments and decrease in 5% treatment. The results of the average test show that there is an increase in the water absorption value of the thermoplastic film which is proportional to the increase in the addition of cellulose. Thermoplastic film has optimal water absorption at the addition of 5% cellulose, namely 70.44 (Figure 4).

Cassava starch used in the manufacture of films contains high amylose which has an effect on increasing the strong film structure. The use of starch can also produce low transparency properties, brittleness, hydrophilic properties causing a low water vapor barrier thereby affecting the stability and mechanical properties of the film. Glycerol is one of the film-forming materials in this study where the effect of plasticizers can reduce the solubility of thermoplastic films so that the resistance to water is high which is proportional to the increase in the film matrix so that the smaller the value produces a film structure that is strong and not easily destroyed by water. The higher the plasticizer concentration, the lower the solubility in water. Glycerol is a plasticizer that is soluble in water and alcohol in which the molecules are relatively small hydrophilic and form hydrogen bonds. Glycerol has the ability to increase water binding to the film (Sitompul & Zubaidah, 2017). Based on the results of the ANOVA test value of variance, the value of 0.982 had no significant effect on film solubility, so further tests were not necessary.

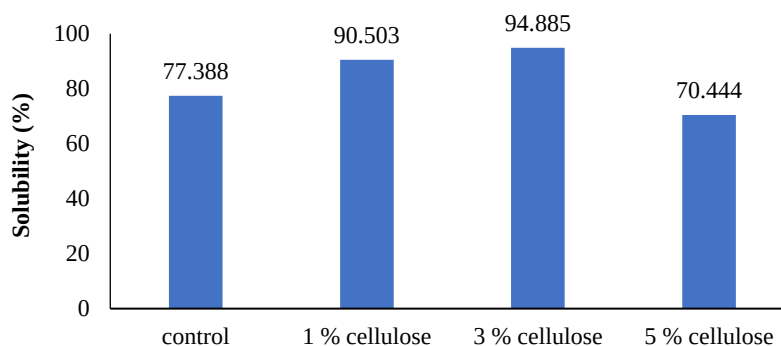


Figure 4. Solubility of film starch resulted from different treatments

3.4. Density

Thermoplastic films have the property of easily melting at a certain temperature, this is because the basic ingredients in making films are biopolymer-based so they are reversible. The lower the density value of a film, the film has an open structure and is easily penetrated by water, CO₂ and oxygen. Conventional films usually have a high value. The density value of the thermoplastic film from coconut coir can be seen in Figure 5. It can be observed that increasing starch content has decreased the bioplastic density. Statistical analysis using ANOVA revealed that the decrease of density due to starch content is highly significant with p value of <0.01 .

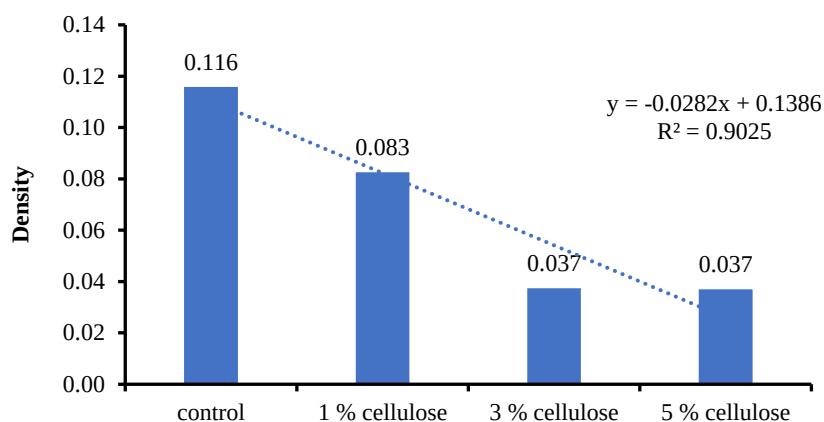


Figure 5. Density of film starch resulted from different treatments

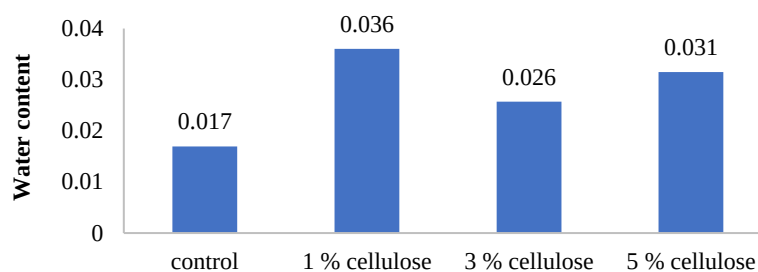


Figure 6. Water content of film starch resulted from different treatments

3.5. Water content

The water content in the film affects the quality of the film because it will affect the quality of product storage. The water content of the film will be affected by the material content of the film. The water content in the product is known by calculating the percentage difference between the start of the test and the end of the test. The test results can be seen in Figure 6. In the figure it can be seen that the least water content is the 3% treatment with a value of 0.026 when compared to the control which has a value of 0.017. Based on the ANOVA test, it showed a sig value of 0.649 which stated that biofilm had no significant effect. This was because the cellulose fiber used was hygroscopic, so it would absorb water if used in large quantities. The higher the percentage of fiber used, the greater the ability to absorb free water contained in the film.

3.6. Chemical Characteristics

FTIR spectrum analysis was carried out to determine the value of functional groups contained in coir, delignified coir and coir cellulose. The FTIR spectrum of coir, delignified coir and cellulose can be seen in Figure 7. The characteristics of the functional groups contained in coconut fiber, delignified coconut fiber and cellulose can be seen in Figure 6. Changes in the chemical composition and structure of coir, coir during the delignification process and coir cellulose are identified by functional groups that have the ability to absorb water vapor. The transmittance curves have different

shapes. The most obvious difference is shown in the husks. The peak intensity of husk is shown at wave numbers 3400, 2700, 1600, 1255, and 1029 cm^{-1} . The broad peak intensity at a higher frequency, namely 3400 cm^{-1} , indicates the presence of OH groups in the coir. According to Johar *et al.* (2012) at wave numbers between 3440-3400 cm^{-1} shows the presence of O-H and C-H groups. The peak at wave number 2700 cm^{-1} indicates the stretching of OH groups attached to methyl groups which are commonly found in lignin (Tarley & Arruda, 2004). The peak at a frequency of 1600 cm^{-1} shows the material's ability to absorb water due to stretching of hydrogen bonds and OH group bonds in the cellulose structure (Johar *et al.* 2012). The decrease in the peak at around 1255 cm^{-1} in activated coir indicates changes in lignin levels. This indicates stretching of the bonds in the C=C symmetric aromatic groups in lignin (Khalil *et al.* 2018). Changes in the structure of cellulose occur at a frequency of 2775 indicating the loss of carboxylic acid groups with OH bonds after alkaline treatment and the loss of amide groups (N-H) at a frequency of 3500-3700 cm^{-1} . The SiO-Si group was found in delignified coir at a frequency of 1026 cm^{-1} . A frequency of 700 cm^{-1} indicates the presence of a hydroxyl group (OH). Increasing the number of hydrophilic sites increases the viability of water absorption. The FTIR spectrum of cellulose isolation shows a peak wavelength of 3400 cm^{-1} in the presence of O-H (hydroxyl or alcohol) groups from cellulose which absorb water. The C=C group which identifies lignin is found at a wavelength of 1600 cm^{-1} , this can be seen from the change in coir waves with delignified coir. At this wavelength there are aromatic rings as an indication that lignin is still present (Yang *et al.*, 2007).

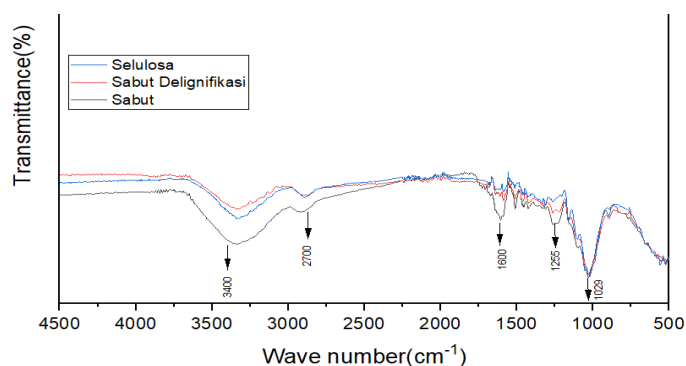


Figure 7. FTIR results of coir, delignified coir, coconut fiber cellulose

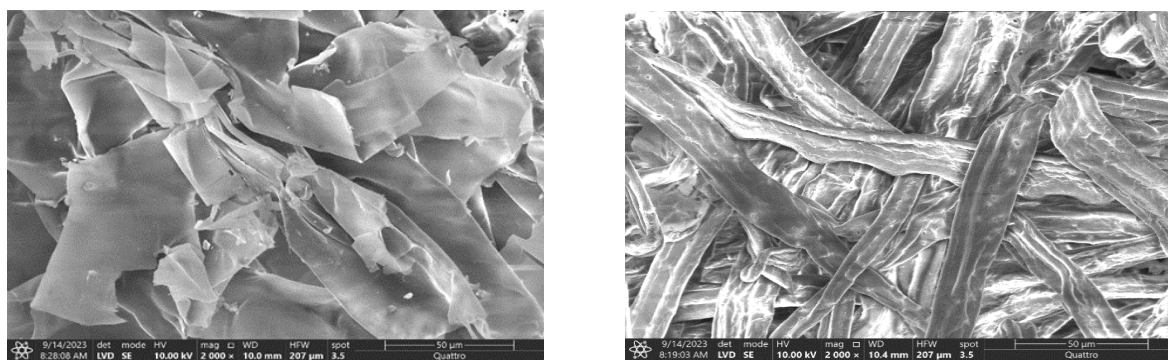


Figure 8. SEM of film: head coat (left), and coconut fiber cellulose (right)

3.7. Scanning Electron Microscope

The characteristics of the adsorber matrix can be seen from the surface condition of the coconut fiber from the condition it is still in the form of coconut fiber and after it becomes coconut fiber cellulose fiber. In this test the coating used was a gold coating, the coating was carried out under vacuum conditions. After the vacuum condition is reached, the scanning process can be carried out by activating the electron beam (Javed *et al.* 2008). The scanning process was carried out with 1000X magnification (Figure 8). The differences between the two coconut fibers in the conditions before and after

becoming coconut fiber cellulose fiber. In the process of making cellulose fiber, the coir first goes through a delignification process which causes the coir structure to become layered and sloppy without damaging its inherent structure (Emdadi *et al.*, 2014). While in Figure 8b is husk fiber that has undergone a bleaching process with H_2O_2 solution. The morphology of coconut fiber which has become cellulose in Figure 8 looks cleaner and has a more regular structure which can be seen from the density and neatness. This is due to the hydrogen bonds and cell wall structure being broken and a small and regular material being formed (Siró & Plackett, 2010).

3.8. Color Change (Visual)

The cellulose film application visual shows that chilies that are packaged in TPS packaging and stored at room and cold temperatures, experience changes in shape and color which can be seen as the longer they are stored, the drier they become. However, chilies that are stored at cold temperature experience changes in color and shape that are different from room temperature, as can be seen in Figure 9. For color, in the picture above you can see that chilies stored at cold temperatures have a fresher and brighter color until the last day of storage. For texture and shape, at room temperature there has been a change on the 7th day of storage, until the last day of storage on the 19th day the chilies stored at room temperature have been very dry, different from the chilies packaged with TPS film and stored at cold temperatures, still has good texture and good shape is not much different from the fresh harvest. This is because temperature can influence metabolism and control ripeness thereby extending the shelf life of fruit (Paramita, 2010). Using TPS film with the addition of coconut fiber cellulose fiber in chili packaging can maintain good quality of chilies, because plastic film can inhibit the rate of respiration by suppressing ATP-ase activity in producing energy that will be used during the respiration process.

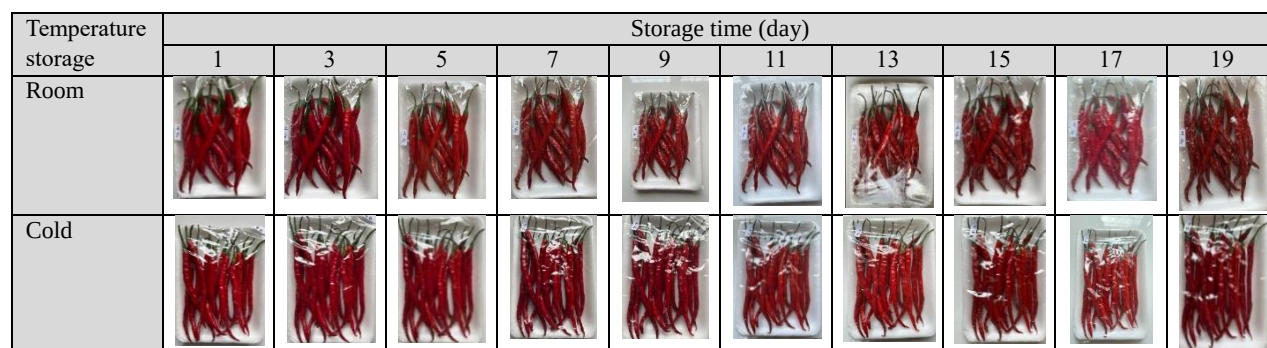


Figure 9. Visual changes in chili during storage using bioplastic film for 19 days

3.9. Weight Loss

The application of thermoplastic film in this research uses the best results from previous research by Sari *et al.* (2023) with the formulation of adding 5% w/v coconut fiber cellulose to cellulose thermoplastic film. Along with the long storage time, weight loss increases quite significantly from day to day. The weight loss that occurs in red chili samples during storage can be seen in Figure 10. The use of cellulose film applied to red chilies was carried out at two temperatures, namely room temperature (25-27 °C) and cold temperature (4-10 °C). The effect of storage using cellulose film at room and cold temperatures in Figure 10 Thermoplastic cellulose (TPS) can be seen to increase during 19 days of storage. The increase in weight loss during storage is very visible at room temperature, this is because temperature greatly influences the activity of the water content in the chili samples used. The higher the temperature, the faster the loss of water content and other substances in the sample. In contrast to low temperatures which can retain water vapor and other substances.

Figure 10 shows the effect of sample treatment using TPS film on sample weight loss during 19 days of storage. Application at room temperature is known to produce the highest weight loss values when compared to cold temperature treatment. The smallest weight loss was produced by chili samples stored at cold temperatures with an average weight loss during 19 days of storage with a weight loss of 0.42 g. In the room temperature treatment, it was found that on day

19 the weight loss was 1.06 g. Based on the results of the hypothesis test with the best treatment from previous film making that had been carried out, the TPS film applied to chilies, for weight loss, was not significantly different, showing a calculated F statistical value of 2.45 with a level of 5%. Disruption of metabolic processes causes minimal loss of product water so that the resulting weight loss during storage is relatively small. Weight loss during storage is influenced by the respiration rate which causes the loss of a certain amount of carbon that occurs during the ripening process (Brackmann *et al.*, 2014). However, the main factor that causes weight loss is the loss of water vapor that occurs during the storage process. Fruit weight loss is related to loss of water vapor and the withering process which is closely related to the high metabolic rate that occurs (Vázquez-Celestino *et al.*, 2016). Chili is a climacteric plant with a relatively fast respiration rate, this condition can indicate a high metabolic rate which has the potential to cause a greater rate of water vapor loss.

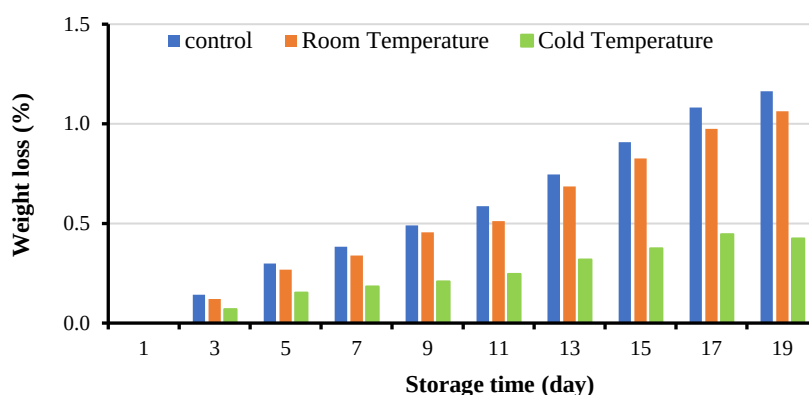


Figure 10. Weight loss of red chilies during storage for 19 days

3.10. Hardness

The level of ripeness in chilies is also indicated by the hardness test parameter (Zerbini *et al.*, 2015). The decrease in hardness value occurs due to changes in cell walls during the cooking process. The level of hardness is one of the parameters that must be considered during storage (Ali *et al.*, 2014). This is because a decrease in the level of hardness or softness of the texture will cause the rotting process to occur and increase the potential for damage to horticultural products during distribution. Figure 11 shows the effect of storage with the addition of cellulose starch thermoplastic film on the texture of chili.

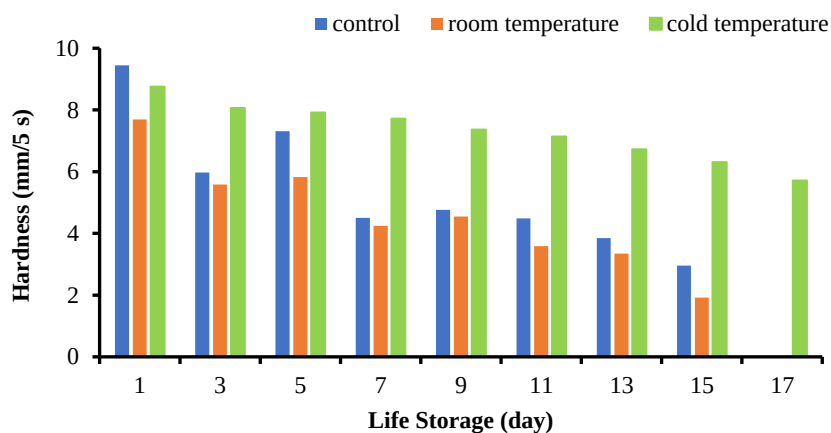


Figure 11. The level of hardness of chilies during storage

Figure 11 shows the hardness value of chilies during 17 days of storage. The longer the storage, the softer the chilies will be. The control was found to produce the highest measurement value when compared to the room temperature treatment and the cold temperature treatment had a relatively stable level of hardness until the 17th day of storage. For control, on the first day of storage, the hardness value was 9.45 mm/5 seconds and on the last day of storage, on day 15, it had a hardness value of 2.96 mm/5 seconds. This is different from cold temperature storage, where on the first day of storage it was 8.75 mm/5 seconds and the sample was able to survive until the 17th day with a value of 5.71 mm/5 seconds. The decrease in hardness value occurs due to the breakdown of cell wall components consisting of cellulose, hemicellulose, pectin and lignin, pectinic acid and pectic acid so that the fruit becomes softer. The ripening process causes a reduction in the firmness of horticultural products, this is due to the conversion of protopectin into pectin. Based on the statistical results of the research carried out, the addition of coconut fiber cellulose to thermoplastic film and applied to packaging for storing chilies was able to provide an effect that was not significantly different to the level of 0.561 on changes in the hardness of chilies.

4. CONCLUSION

The results of the identification of the quality of thermoplastic biofilms made from coconut coir on the characteristic test, the film contains cellulose from coconut coir as a reinforcing agent for the film composite on the biodegradation properties of the films produced. It is known that the addition of 5% w/v coco cellulose fiber is the best yield formulation when compared to 1% and 3% w/v formulations.

The thermoplastic biofilm made from 5% w/v coconut fiber cellulose applied to the storage of chili horticultural products are known to be able to maintain the quality of chilies at cold temperatures for 19 days with the quality of the chilies still being fresh when compared to chilies stored at room temperature. In terms of visual changes, chilies stored at room temperature experienced changes in texture and shape on the 9th day of storage.

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