

Synthesis of Film Cellulose-Glycerol-Cloves Oil as Chili Packaging

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ABSTRACT

The aim of this research is to know the utilization of cellulose film with the addition of glycerol and clove oil in various variations as the best chilli packer based on mechanical properties, surface photo, water resistance test, and its effectiveness to extend the life of chilli. The production of cellulose film as packaging in this research is done by using coating method. In the coating method, variations of glycerol and clove oil were added in the ratio of (3: 1), (5: 1), (7: 1), (10: 1), and (15: 1) (v / v). The synthesized cellulose film will be characterized by functional group analysis using FTIR-ATR, mechanical test using Tensile strength, surface photo using optical microscope, cellulose film resistance test to water, and effectiveness test as chili packaging. FS Gli: Ck (3: 1) is a cellulose film with variations in the addition of glycerol and the best clove oil which has a tensile strength of 12.193 MPa, elongation of 9.761%, the most homogeneous surface photo, the greatest water resistance value 42,87, and effectiveness best cellulose film as a chilli packer.

Keyword: : cellulose film, glycerol, clove oil, coating method, chili packaging film

1. INTRODUCTION

Thin layers, known as films, have the potential to serve as coatings for various products, including food items. These films are particularly valuable for packaging materials, as they can function as a barrier against the transfer of mass, which includes water content, oxygen, fat, and light. Films can be made from agricultural polymers such as cellulose, for example cellulose from nata de coco, chitin (in the skin of crustaceae) and pullulan (the result of starch fermentation by pullularia pullulans) (Coniwanti, Laila & Alfira, 2014). In this study, the film was made from nata de coco cellulose. Cellulose from nata de coco is a cellulose fiber produced by bacteria or called bacterial cellulose (BC). Bacterial cellulose has good liquid absorption, has a high water content, and can be sterilized without affecting the characteristics of the material (Rohaeti, Widjajanti & Rakhmawati, 2016). The process of making nata is done through several stages, namely Maintenance of pure culture Acetobacter xylinum, starter production, fermentation, and harvesting (Nurlinda, Darmawati & Mahadi, 2013).

Cellulose films from nata de coco have poor mechanical properties, which have a stiff texture. Therefore, it is necessary to add plasticizer additives to improve its mechanical properties. In this study, glycerol will be added as a plasticizer. As a plasticizer glycerol can reduce intermolecular hydrogen bonds and increase film mobility resulting in enhanced film flexibility and

extensibility. This causes the stiffness level of the film to decrease (Radhiyatullah, Indriani & Ginting, 2015).

In addition to glycerol, clove oil is used in the production of cellulose films for chili packaging. The main component in cloves is eugenol with a percentage of 70-80%. Eugenol is an active ingredient to kill bacteria (Ajiboye, Mohammed & Bello, 2016). Clove oil also has biological activities, including antibacterial, antifungal, insect repellent, and antioxidant properties (Andries, Gunawan & Supit, 2014). With clove oil's antibacterial activity, it is believed that the cellulose film created by it will extend the longevity of the product if the cellulose film is later used as packaging.

The purpose of this study was to determine the effect of the addition of glycerol and clove oil on the mechanical properties of cellulose films. The mechanical properties observed were tensile strength and percent elongation.

2. RESEARCH METHOD

2.1 Materials

Waste coconut water, granulated sugar, ZA food solids, nata seed solution containing *Acetobacter xylinum* bacteria, Merck 5% HCl solution, glycerol, distilled water, and clove oil.

2.2 Tools

Glasswares, Petridish, pH meter, vacuum pump, hot plate and stirrer, analytical balance, one ASTM D638 tensile strength measuring device, optical microscope namely nikon imagine system microphotography, Shimadzu IR FTIR-ATR spectrophotometer.

2.3 Production of Nata de Coco

The production of nata de coco begins with preparing 1000 mL of coconut water. Sugar as much as 10% (b/v) and ZA food as much as 0.5% (b/v) were added to the coconut water. The solution was heated to boil. After the solution cooled, the pH of the solution was measured, if the pH was less than 4, then the solution was added with acetic acid until the pH was around 3-4. The solution was then poured into the fermentation container. Furthermore, 20% (v/v) *Acetobacter Xylinum* was added and the solution was rested for 4 days until a thin layer of nata was formed. The nata de coco sheet that has been successfully made was then boiled, pressed and dried for 2 days under the sun, after drying the nata sheet (FS nata) was mashed by cutting it into very small pieces. Figure 1 shows a successfully prepared nata de coco.



Figure 1. The produced nata de coco sheet

The sheets of nata de coco obtained in this study were white in color. Furthermore, the properly made nata de coco had a chewy texture with a thickness of about 0.4-0.5 cm.

2.3 Production of Cellulose/Glycerol (FS+Gli), Cellulose/Clove Oil (FS+Ck), and Cellulose/Glycerol + Clove Oil (FS Gli:Ck) Films by Coating Method

During the production of FS + Gli film, an amount of 15 ml of glycerol plasticizer was added. Similarly, in the production of FS + Ck film, a quantity of 15 ml of clove oil was added. While in the production of FS Gli film, the addition of glycerol and clove oil was added with varying ratios of glycerol to clove oil. These ratios included 3:1, 5:1, 7:1, 10:1, and 15:1 (v/v). The application of the solution onto the film was conducted by pouring it over the nata de coco sheet until its full coverage. The soaking process was subsequently carried out for 24 hours. Subsequently, the film was subjected to a drying process under sunlight for an estimated duration of 2 days.

The following are definitions of used abbreviations:

FS = Cellulose Film

Gli = Glycerol

Ck = Clove Oil

3. RESULTS AND ANALYSIS

The current study produced a cellulose film that exhibits a thin, slightly transparent sheet with a smooth surface texture. Upon manual testing, the film has demonstrated a notable degree of elasticity. Figure 2 shows the cellulose film created in this study.



Figure 2. Cellulose Film with Prepared Glycerol and Clove Oil

The mechanical properties of the cellulose film were evaluated through the application of a tensile strength test and percent elongation test, in accordance with the guidelines outlined in ASTM D638. Furthermore, the functional groups in cellulose film were analyzed via Fourier Transform Infrared Attenuated Total Reflectance (FTIR-ATR) spectroscopy. In addition, an optical microscope was utilized to conduct a surface morphology analysis of the cellulose film.

3.1 Effect of Glycerol and Clove Oil Addition on Tensile Strength of Cellulose Film

Figure 3 illustrates the effect of glycerol and clove oil addition on the tensile strength of cellulose sheets.

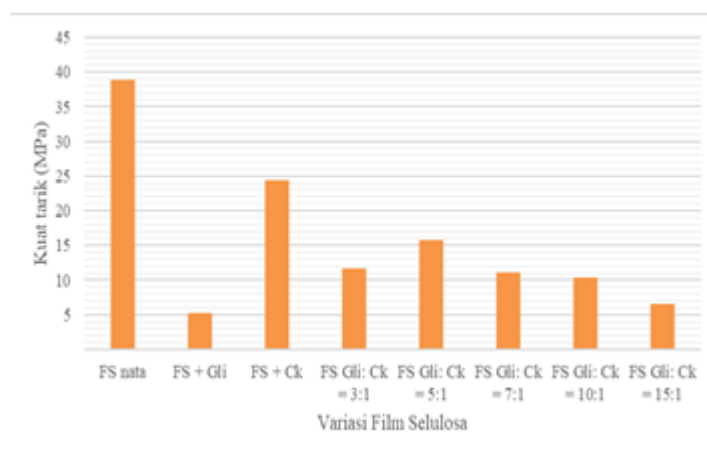


Figure 3. Effect of Glycerol and Clove Oil Addition on Tensile Strength of Cellulose Film

According to Figure 3, the tensile strength of the film reduces when glycerol and clove oil are added when compared to cellulose nata film (FS nata). Furthermore, differences in the amounts of glycerol and clove oil added to cellulose films result in different tensile strength values. In general, the addition of glycerol and clove oil increases the tensile strength value of cellulose films from (3:1) to (5:1) to (7:1). Tensile strength values decreased in cellulose films with the addition of glycerol and clove oil in the ratios of (7:1) to (15:1). FS Gli: Ck (1:5) has the maximum tensile strength rating of 13.637 MPa.

Glycerol plasticizers can potentially flex the polymer matrix. The polar group (-OH) in glycerol will cause the polymer bonds in the film to expand so that the intermolecular bonds in the cellulose film will decrease and consequently the tensile strength value of the film will decrease (Huri & Nisa, 2014). The addition of clove oil to cellulose films can reduce tensile strength values. The dispersion of clove oil in the film increases steric hindrance, causing intermolecular interactions in cellulose films to weaken. The decreased intermolecular connections might reduce the tensile strength of the film, resulting in an easily broken film.

3.2 Effect of Glycerol and Clove Oil Addition on Percent Elongation of Cellulose Film

Figure 4 depicts the effect of glycerol and clove oil addition on the percent elongation of cellulose films.

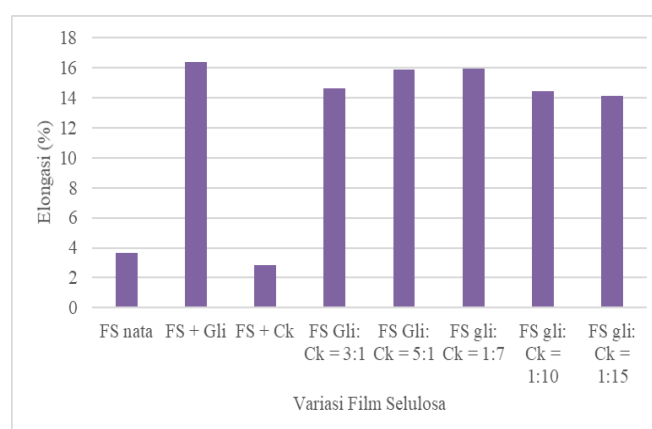


Figure 4. Effect of Glycerol and Clove Oil Addition on Percent Elongation of Cellulose Film

According to Figure 4, the addition of glycerol and clove oil to cellulose films causes the percent elongation obtained to vary as well. In accordance with the graph, FS Gli: Ck cellulose film (3:1) with a percent elongation of 14.630 enhanced the percent elongation of FS Gli: Ck (5:1) to

15.8623. Furthermore, the percent elongation value of the cellulose film of glycerol: clove oil in the ratios of (5:1), (7:1), (10:1), and (15:1) dropped. The percent elongation values of the four cellulose films were respectively 15.8623, 15.9504, 14.4254, and 14.1282. FS Gli: Ck (5:1) has the largest percent elongation, whereas FS Gli: Ck (15:1) has the lowest percent elongation.

When glycerol is added to cellulose films, the intermolecular forces of the cellulose films decrease. The decrease in intermolecular pressures on the film increases its flexibility and extensibility, increasing the percent elongation with the addition of glycerol volume (Radhiyatullah, Indriani & Ginting, 2015). The addition of clove oil to cellulose films might also induce a decrease in percent elongation because the added clove oil will be distributed into the film matrix, causing the intermolecular distance in the film to decrease. The elongation value can decrease when intermolecular links weaken. (Safitri, Riza & Syaubari, 2016).

3.3 Characterization of Cellulose Film Functional Groups

The functional groups of the produced cellulose film were determined by FTIR-ATR. Figure 5 illustrates the outcomes of this characterization.

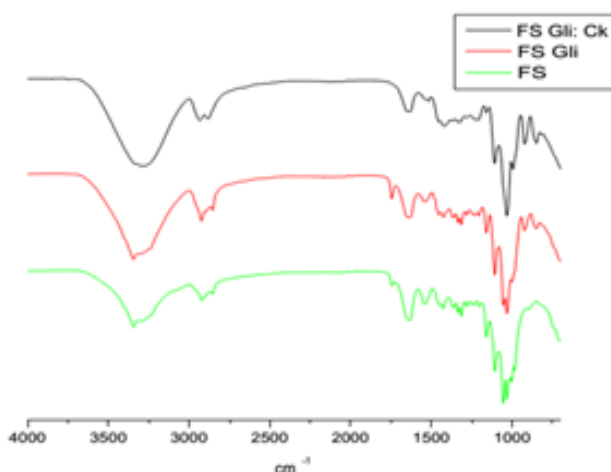


Figure 5. Results of Function Group Analysis of Cellulose Film

The FTIR-ATR spectra of FS nata, FS + Gli, and FS Gli: Ck are almost identical and do not reveal substantial differences in the resultant spectra. However, there are shifts and variances in OH absorption visible in the spectra. The change in absorption occurred because OH absorption showed at 3344.98 cm^{-1} in (FS nata), but at 3346.10 cm^{-1} and 3278.05 cm^{-1} in (FS + Gli) and (FS Gli: Ck). Aside from the shift in OH absorption in the three films, the cellulose film with the addition of glycerol and clove oil (FS Gli: Ck) has the greatest OH absorption. The expansion happens due to interactions between cellulose, glycerol, and clove oil caused by the addition of glycerol and clove oil to the cellulose film. The interaction expanded the OH spectrum on the FS Gli: Ck spectrum. Furthermore, the presence of intermolecular hydrogen bonds in the molecule can cause the OH absorption to broaden (Pavia et al, 2010). The addition of glycerol and clove oil, both of which include an OH group, enables the FS Gli: Ck OH spectrum to become sharper and stronger (Setiani, Sudiarti & Rahmidar, 2013).

3.4 Surface Photo of Cellulose Film

Figure 6 exhibits the outcome of the cellulose film surface photo.

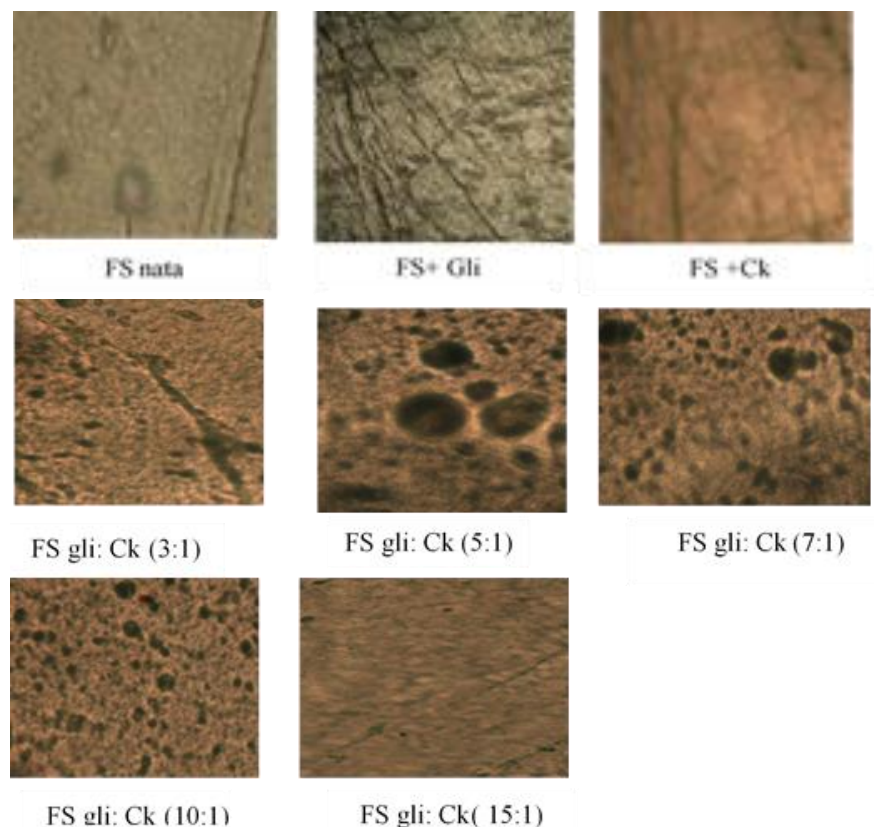


Figure 6. Photograph of Cellulose Film Surface Results

An optical microscope with a magnification of 40 times was used to perform photo characterization of the film surface. The cellulose nata film (FS nata) surface photo has a flat surface structure. While cellulose films with glycerol added (FS + Gli) and cellulose films with clove oil added (FS + Ck) have an irregular surface structure due to the presence of spots on the surface.

The surface morphology of cellulose films varies due to variations in the addition of glycerol and clove oil in cellulose filmmaking. The more changes in glycerol and clove oil added, the more dots on the film's surface. The spots appear because the glycerol and clove oil are not completely dissolved and disseminated on the cellulose sheet. A film with a more homogenous surface will result from the proper addition of plasticizers.

4. CONCLUSION

The mechanical properties of cellulose films are affected by the addition of glycerol and clove oil. Variations in the addition of glycerol and clove oil to cellulose films result in different tensile strength and percent elongation values that fluctuate.

The finest mechanical properties of cellulose films with the addition of glycerol and clove oil are obtained from cellulose films at a 5: 1 ratio (FS Gli: CK = 5: 1), with a tensile strength value of 13.637 MPa and an elongation value of 15.499%. The FTIR study revealed no significant differences in the spectra before and after the addition of glycerol and clove oil. Surface photos revealed that the combination of glycerol and clove oil caused the cellulose film's surface to become uneven.

REFERENCES

- Ajiboye, T. O., Mohammed, A. O., Bello, S. A., et al. (2016). Antibacterial Activity of *Syzygium Aromaticum* Seed: Studies on Oxidative Stress Biomarkers and Membrane Permeability. *Microbial Pathogenesis*, 95,208–15. <http://dx.doi.org/10.1016/j.micpath.2016.03.011>).

- Andries, J.R., Gunawan, P.N. & Supit, A., (2014). Uji Efek Anti Bakteri Ekstrak Bunga Cengkeh Terhadap Bakteri *Streptococcus Mutans* secara In Vitro. *E-GIGI* 2. (<http://ejournal.unsrat.ac.id/index.php/egigi/article/view/5763>).
- Coniwanti, P., Laila, L., & Alfira, M.R. (2014). Pembuatan Film Plastik Biodegradabel Dari Pati Jagung Dengan Penambahan Kitosan Dan Pemplastis Gliserol. *Jurnal Teknik Kimia*, 4, 22–30.
- Huri, D. & Nisa, F.C. (2014). Pengaruh Konsentrasi Gliserol dan Ekstrak Ampas Kulit Apel terhadap Karakteristik Fisik dan Kimia Edible Film. *Jurnal Pangan dan Agroindustri*, 2, 29–40.
- Nurlinda, Darmawati, Mahadi, I., (2013). The Effectiveness Sugar Addition of Nata de Cassava Quality from Tapioca Liquid Waste As Learning Module Development on Conventional Biotechnology Concept Grade XII Senior High School. Study Program of Biology Education, Faculty of Teacher Training and Education University of Riau." 1–11.
- Pavia, D.L., Lampman, G.M., Kriz, G.S., et al (2010). *Introduction to Spectroscopy*. United States of America: TSI Graphics.
- Radhiyatullah, A., Indriani, N., Ginting, M.H.S., (2015). Pengaruh Berat Pati dan Volume Plasticizer Gliserol Terhadap Karakteristik Film Bioplastik Pati Kentang. *Jurnal Teknik Kimia*, 4, 35–39.
- Rohaeti, E., Widjajanti, E., Rakhmawati, A., (2016). Kemudahan Biodegradasi Selulosa Bakteri dari Limbah Cucian Beras. *Jurnal Kimia VALENSI: Jurnal Penelitian Dan Pengembangan Ilmu Kimia*, 1, 35–44.
- Safitri, I., Riza, M. & Syaubari, (2016). Uji Mekanik Plastik Biodegradable dari Pati Sagu dan Grafting Poly (Nipam) -Kitosan dengan Penambahan Minyak Kayu Manis (*Cinnamomum burmannii*) sebagai Antioksidan. *Jurnal Litbang Industri* , 6, 107–116.
- Setiani, W., Sudiarti, W., Rahmidar, L., (2013). Preparasi dan Karakterisasi Edible Film dari Poliblend Pati Sukun-Kitosan." *Valensi*, 3, 100–109.