

The Potential of Cellulose-Glycerol-Lemongrass Oil Films

Running Title: As an Eco-friendly Packaging Materials

Endang W. Laksono^{*}, F. M. Cahyani, Dewi Yuanita, Isana SYL

Department of Chemistry Education, FMIPA Universitas Negeri Yogyakarta, Indonesia

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Abstract One of the big problems in Indonesia currently is waste management, especially plastic waste. Plastic that comes from food packaging is a source of environmental problems because it is difficult to decompose naturally. Therefore, it is necessary to look for alternative materials to replace petroleum-based plastic packaging, with other materials based on renewable and biodegradable materials. This study aims to determine the properties of cellulose films with the addition of glycerol and lemongrass oil based on the characterization of mechanical tests, water and vapor resistance tests, and functional groups using FTIR-ATR spectrophotometer and the possibility of their use as packaging. Cellulose film was fermented from coconut water and coated sequentially by glycerol (5,10,15 and 20 % w/w) and lemongrass oils. Coating method with lemongrass oil was done for 0, 1, 3, 5, 7, and 14 days. The cellulose film was characterized using tensile strength, water resistance test, water vapor test and FTIR-ATR. The results showed that glycerol and lemongrass oil can affect the physic and mechanic cellulose film. The cellulose film with a concentration of glycerol of 5% and coated by lemongrass oil for 5 days has the best elongation and modulus Young 230.39 MPa. The cellulose film with concentration of glycerol 5% and coated by lemongrass oil for 14 days has the best water vapor resistance and water resistance value of 88.298% and 49.93 respectively and shows an increase in function group intensity of C=O. Thus, the film of cellulose-glycerol-lemongrass is an alternative packaging material.

Keywords Cellulose Films, Lemongrass Oil, Packaging Materials, Modulus Young

1. Introduction

Packaging is very important in life because it is a protector of a product. Products that really need packaging is food. The main role of food packaging is to protect food products. Food packaging can retard product deterioration, retain the beneficial effects of processing, extend shelf-life, and maintain or increase the quality and safety of food [1,2]. In doing so, packaging provides protection from 3 major classes of external influences: chemical, biological, and physical. The times and technology encourage people to use a lot of packaging from synthetic materials, one of which is plastic. The type of plastic that is widely used is synthetic plastic from polymers with non-renewable petroleum raw materials and the amount is limited [3], especially if the plastic waste is burned, of course it will produce gases that are harmful to humans [4]. The waste of using plastic can damage the environment, so plastic needs to be replaced with other materials that have a smaller negative impact. Public's awareness of the environment and healthy food is getting higher, making it possible to receive plastic's replacement. One alternative is to use films made of environmentally friendly materials as packaging, for example film of nata de coco. Film can be

an alternative to packaging because it is biodegradable and at the same time acts as a barrier to control the transfer of water vapor, lipids and oxygen. Film is a biopolymer which can be decomposed and divided into three types based on its constituent, that are hydrocolloid, fat, and composites [5,6]. Nata is a pellicle or microfibril network, namely cellulose which has a chemical structure like that of higher plants [7]. Films can be added with additives to improve quality, texture, aroma, and color, and they can also be used to control microbial activity.

Cellulose film of the hydrocolloid type is an alternative to plastic because it is more environmentally friendly. The source of cellulose in this study is nata de coco. Nata de coco is fermented of coconut water with the help of the *Acetobacter Xylinum* bacterium as a starter. In general, nata is cellulose which is a fibril mass that resembles an irregular thread when viewed under a microscope. The mechanism for the formation of bacterial cellulose in nata consists of four stages. The first step is the hydrolysis of sucrose into fructose and glucose, followed by the isomerase step which converts intramolecular α -D-glucose to β -D-glucose. In the third stage, there is a reaction of intramolecular coupling of glucose to form a 1,4- β -glycoside bond. The final step is the polymerization reaction to form bacterial cellulose in nata with its repeat unit, namely cellulose [8]. Based on the process of forming cellulose, bacterial cellulose is pure cellulose. However, cellulose is hygroscopic, hard and brittle [9]. This property is not good when used as packaging, therefore additional materials need to be added so that bacterial cellulose becomes more flexible and elastic. Additional ingredients that can be added include glycerol, polyethene glycol, and sorbitol [10]. The addition of additional materials to cellulose is expected to improve the quality of cellulose films so that they can be used as packaging [11]. Among the plasticizers, glycerol has been used as a plasticizer to produce starch-based films due to its compatibility with amylose, which stimulates better mechanical properties through interference with amylose, packing by reducing the intermolecular forces between starch molecules [12]. Several studies using films coated with glycerol plasticizer [13,14] have shown that the addition of glycerol to cellulose affects tensile strength, which is because glycerol aims to reduce the bonding power of cellulose molecules so that the flexibility of bioplastics increases. Glycerol has the advantage of increasing flexibility, appearance, or morphology, and its mechanical properties do not change during storage [15].

Another effort to increase the excellence of film is by adding lemongrass oils (*Cymbopogon citrus*). Essential oils in lemongrass can be used as inhibitors of microbial growth and their hydrophobic properties cause packaging of products to not be damaged quickly and can extend storage power. One of the goals of packaging is to extend the shelf life of perishable foods, by reducing the movement of particles from packaging materials to food, and eliminating industrial processes that can cause the entry of pathogenic

microorganisms into products [16]. The presence of oxidation processes leads to damage to food. One of the causes is the oxidation of lipids, resulting in changes in color, texture, taste, smell, loss of nutrients and even produce toxins [17]. Since essential oils are rich in antioxidants, they are commonly used in edible films and coatings [18,19]. The antioxidant activity of essential oils can be expressed by their ability to act as oxygen scavengers and allow the diffusion of active agents into coated food products as the studies dealing with the effect of essential oil addition on the *in vitro* antioxidant properties of films [17]. The research by Pires et al. [20], stated that the addition of lemongrass oil to hake proteins edible films will cause a decrease in mechanical properties, and inhibition of *Shewanella putrefaciens*, but increase antioxidant properties.

This study aims to determine the effect of glycerol and lemongrass oil addition on film mechanical tests and to determine the characteristics of cellulose-glycerol films based on characterization using FTIR, homogeneity test, mechanical tests, water resistance tests, and vapor resistance tests.

2. Materials and Methods

2.1. Chemicals and Materials

Fresh coconut water, ammonium sulfate (p.a, Merck), glucose, glycerol (p.a), lemongrass oils, glacial acetic acid (pa, Merck), acetobacter xylinum bacteria as fermentation starter cultures.

2.2. Preparation of Film Cellulose-Glycerol-Lemongrass Oils

Glucose and ammonium sulfate were added to the boiling coconut water, and glacial acetic acid was used to bring the pH of the mixture to 4. Glycerol was added to the mixture in varying concentrations of 5%, 10%, or 15% before being left overnight. Acetobacter xylinum bacteria were added, and the mixture fermented for 5 days to form a film layer. For 1, 3, 5, 7, and 14 days, the films were washed, dried, and then coated with lemongrass oils using varying immersion times. The film is then cured outside in the sun.

2.3. Mechanical Analysis Using Tensile Strength

Using a tensile strength tool (ASTM D882) a mechanical test was carried out, and the information obtained was the tensile strength and elongation of the film, which were analyzed using the equation:

$$\sigma = \frac{F}{A}, \text{ which}$$

Tensile strength of cellulose film

σ = tensile strength (MPa)

F = maximum of load (N)

A = sectional area (mm²)

Tensile strength (MPa) was expressed as a ratio of maximum load for breaking the film to initial cross-sectional area of the sample [21]. Meanwhile, percentage of elongation (EAB, %) was expressed as a ratio of the elongation at the break point to the initial length of the sample multiplied by 100.

$$\text{Elongation, } \varepsilon = \frac{L_t - L_o \text{ (mm)}}{L_o \text{ (mm)}} \times 100\%$$

ε = elongation (%)

L_t = extension at break (mm), L_o = initial length (mm)

$$\text{Modulus Young, } E = \frac{\sigma}{\varepsilon}$$

2.4. Analyses of Cellulose Films' Water Resistance and Vapor Permeability

Using the tea bag [22] modification method, information on water resistance was obtained. The swelling test, which measures the percentage at which the film can expand due to the entry of water during immersion at room temperature, can be used to estimate a material's resistance to water. The equation is used to search for the percentage of swelling:

$$\% \text{ swelling} = \frac{W - W_o}{W_o} \times 100\%, \text{ } W_o = \text{dry sample weight}$$

W = sample weight after immersing.

Water resistance is determined by repeating the test 10 times. And the resistance to water can be calculated by the equation = 100% - % swelling.

The cellulose film used for packaging is a moisture resistance material. The amount of film that does not absorb water vapor is used to calculate the vapor permeability value. The method for determining the vapor permeability value is similar to determining the swelling test, but the sample is not immersed in water. The samples were hung on a rope above the water at a safe distance from the water (approximately 1 cm) for 24 hours to measure water vapor absorption. The sample is weighed again after 24 hours have passed so that the percentage of the sample that does not absorb water vapor can be determined. The treatment was carried out with 10 repetitions.

2.5. Fourier Transform Infrared (FTIR) Spectroscopy

A Nicolet Avatar 360 IR Spectrometer was used to obtain the IR spectra of the films. The film sample was placed onto a metallic slit sample holder. The analysis was conducted in the wavenumber range of 400 to 4000 cm⁻¹

with over 10 cumulative scans and 2 cm⁻¹ resolution.

3. Result and Discussion

The dried nata are cellulose films that had been successfully made (Figure 1).

The color of dried nata are white to brown depending on the thickness of the nata. Cellulose films have a strong and elastic texture, insoluble in water, films are transparent, and do not smell.

Table 1 displays the complete physical appearance of the cellulose-glycerol film. Based on Table 1, it appears that the addition of glycerol affects the physical appearance of the film, especially the transparency of the film. With regard to its use as a wrapper, the expected physical appearance is non-transparent, insoluble in water and has a strong texture. As for the color, it can be adjusted with coloring. Transparency and opacity properties are also important parameters that determine the suitability of biopolymers as food packaging materials. High transparency and low opacity values are desired to increase the chances of consumer acceptance of new biomaterials. Research of Vázquez M., and Velazquez G., et al. [23, 24] shows that increasing the glycerol content in bacterial cellulose will increase transparency and reduce opacity. Glycerol interacted with the cellulose polymer chains and forming new interactions. The polymers of glycerol interactions increased the intramolecular distance in the structure, making it less dense, which makes it more transparent [23].

The occurrence of color lightening with the addition of 5-10% glycerol, then darkening again with the addition of 15% glycerol and then brightening again with the addition of 20% glycerol seems to be caused solely by the thickness of the nata.

3.1. Mechanical Properties

Analysis of tensile strength (stress), elongation at break (elongation), and Young's modulus yielded results which became the basis for the mechanical characteristics of cellulose films and cellulose-glycerol films. According to Figure 2, the tensile strength of the film (without glycerol and citronella oil) is greater than 50 MPa. This result turns out to meet the requirements of the Japanese Industrial Standard (JIS) for packing, namely a minimum tensile strength of 40 Kg/cm² or 3.92 MPa [25]. Meanwhile, the Indonesian National Standard (SNI) stipulates a minimum tensile strength of 24.7 MPa for plastic packaging and 0.0016 MPa for paper packaging.

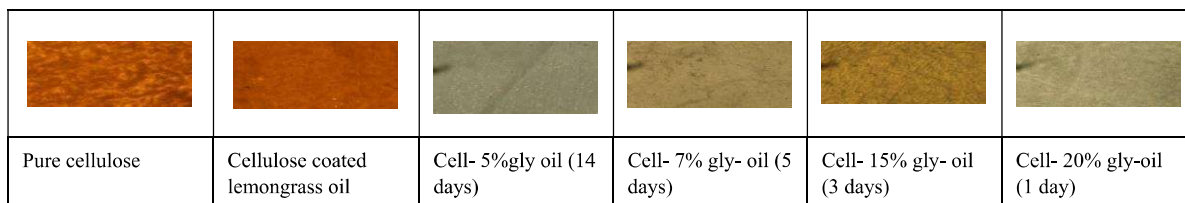


Figure 1. Films of cellulose with glycerol addition and coating with lemongrass oil

Table 1. Physically appearance of cellulose- glycerol

Observed properties	Observation				
	cellulose	+ 5% glycerol	+ 10 % glycerol	+ 15% glycerol	+ 20% glycerol
Color	brown	brown	white	brown	Light brown
Texture	strong	strong	strong	strong	strong
Solubility in Water	none	none	none	none	none
transparency	opaque	opaque	transparent	transparent	transparent

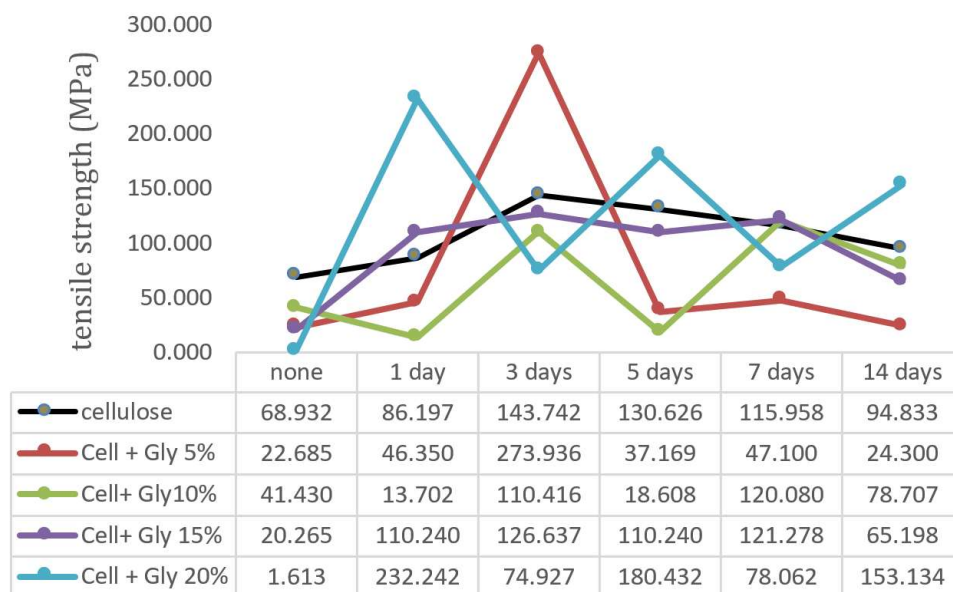


Figure 2. Graph tensile strength of samples

It can be seen in Figure 2 that the inclusion of glycerol molecules reduces the film's tensile strength. By occupying the polymer polysaccharide chains, glycerol molecules will decrease intermolecular connections and weaken the stiffness of cellulose. Glycerol molecules will occupy the polymer polysaccharide chains thereby reducing intermolecular interactions and breaking down the rigidity of cellulose [26]. According to Falguera et al. [27], the tensile strength of the film decreases with the amount of glycerol applied. The solid dispersion system's stability will be decreased by glycerol, and the tensile strength of the polymer chains will be weakened, resulting in a more easily broken film.

The data in Figure 2 shows that the lowest tensile strength is 20% cellulose-glycerol film (without coating

lemongrass oil), which has 1.613 MPa. The high concentration of glycerol makes the film more polar because of its hydrophilic. So, the greater the concentration of glycerol, the lower the tensile strength [28]. But the greatest tensile strength was obtained from samples of cellulose-20% glycerol - soaked in lemongrass oil for 1 day which was equal to 232.24 MPa. Addition of lemongrass oil makes the tensile strength of the film very volatile and unstable due to the hydrophobic of the oil so that it is not perfectly distributed [29]. In general, the tensile strength of the film has met SNI and JIS except for the 20% cellulose-glycerol film without coating by lemongrass oil.

When a film is pulled until it breaks, the length changes by a certain percentage. The elongation demonstrates the film's extensibility. According to SNI (Indonesian National

Standard), plastic must have a minimum of 20% elongation. Figure 3 shows that the cellulose film exhibited an elongation of 7.147% without the addition of glycerol or lemongrass oil.

Glycerol-added cellulose films exhibit a variety of elongations but are typically smaller than pure cellulose films. Because it may bind more OH groups, glycerol can make a film more flexible [30]. With an elongation of 10.741%, cellulose film that had 15% glycerol added had the highest elongation. However, 14 days of immersion in lemongrass oil for films with 15% glycerol added led to an increase in length of 10.416%. This shows that whereas an increase in glycerol concentration ought to improve elongation proportionally, a 20% glycerol concentration

instead has poor elongation. Although the elongation value varies, it is still higher than the elongation of cellulose films that have not been pre-soaked in lemongrass oil. Lemongrass oil and glycerol include citral chemicals that interact with hydrogen bonds to change or increase the film's elongation value [31].

Young's modulus is a measure of the stiffness of a material determined by the ratio between mechanical strength and elongation. Addition of glycerol causes a decrease in Young's modulus significantly. Figure 4, also presents that the 15% cellulose-glycerol film had the lowest Young's modulus of 49.9756 Mpa, much smaller than the Young's modulus of cellulose film.

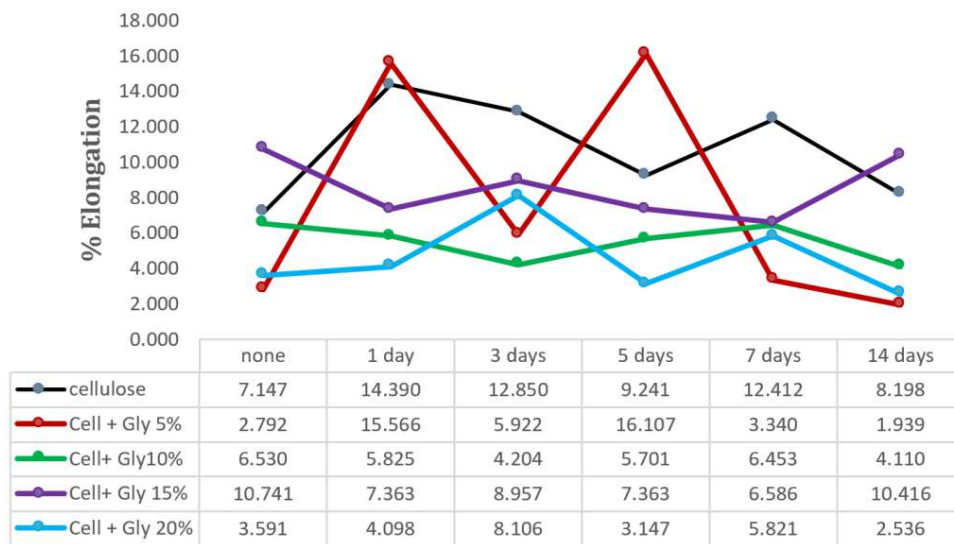


Figure 3. Graph of percentage elongation of samples

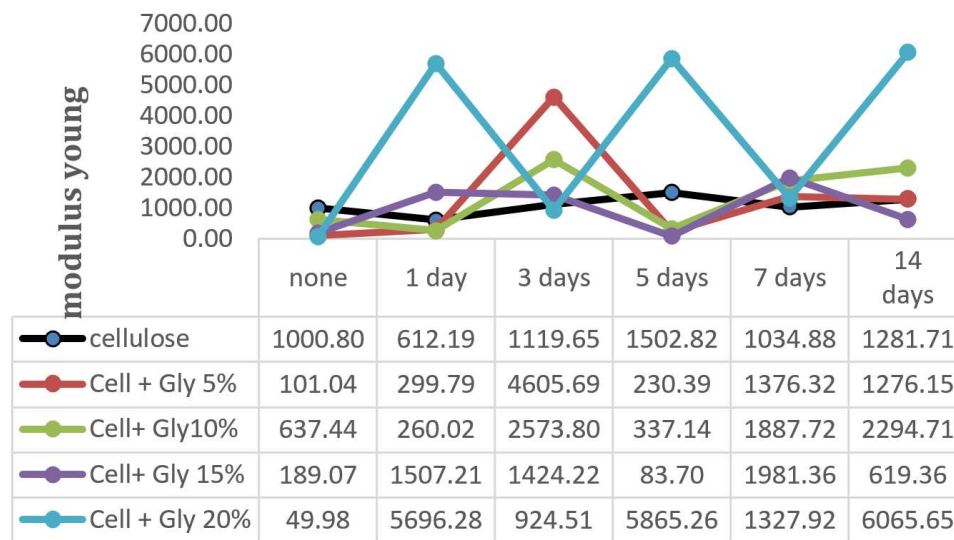


Figure 4. Graph of sample's Modulus Young

The 15% cellulose-glycerol film with 5 day lemongrass oil immersion had the lowest Young modulus with a value of 83.7 MPa. The higher the Young's modulus value, the stiffer the material. The appropriate Young's modulus value is highly dependent on the function of the packaging. If the material is to be used as a box for food, then a material with a high Young's modulus is needed. Conversely, if the packaging is to be used as a substitute for plastic, of course, a material with a relatively low Young's modulus is needed.

3.2. Characterization of Cellulose Films' Water Resistance and Vapor Permeability

The nature of the packaging must be resistant to water and moisture, so that the packaged material is not easily changed or damaged. The relationship between the cellulose, glycerol, and lemongrass oils' water resistance and the amount of glycerol added, as well as the length of time the oils were submerged, is shown in Figure 5. As the amount of glycerol injected increases, water resistance tends to decline. As said by Sudaryanti H.P., [32] glycerol is the simplest glyceride chemical and has a hydroxyl group that is hydrophilic to water and hygroscopic, making it very easy to bond to water. Therefore, water bound to the film will decrease as glycerol concentration decreases, resulting in strong water resistance [33].

The cellulose-glycerol film that has been dipped in lemongrass oil has a higher water resistance rating when compared to cellulose-glycerol films that aren't dyed. This proves that lemongrass oil also affects the cellulose-

glycerol film's water resistance. Being polar and nonpolar, respectively, water and oil do not mix well. The 5% cellulose-glycerol film with a 14-day lemongrass oil soaking has the best water resistance (49.93%), whereas the 20% cellulose-glycerol film with a 1-day lemongrass oil soaking has the lowest (23.15%). The best water resistance means that it can withstand water ingress of up to 49%. In other words, the ability to absorb water from the environment is 51%.

When subjected to a specific water activity or relative humidity (RH) gradient, a package's water vapor permeability (WVP) measures how quickly water vapor travels through the package. The solubility and dispersion of water vapor in a packaging material determine the amount of water vapor permeation. It consists of these three steps: Three processes include water vapor: 1) adsorption on the packaging, 2) diffusion through the package, and 3) desorption from the package. High to low water activity or RH is the primary driver [34].

The procedure is the same as for the water resistance test, but only cellulose and cellulose-glycerol films which have the highest water permeability for each concentration and immersion time are utilized for the vapor permeation test. All films have the best water resistance ratings for a 14-day soaking in lemongrass oil. The vapor permeability of cellulose-glycerol-lemongrass oil is shown in Figure 6. It appears from the figure that cellulose 5% glycerol was immersed in lemongrass oil for 14 days to generate the highest level of permeability.

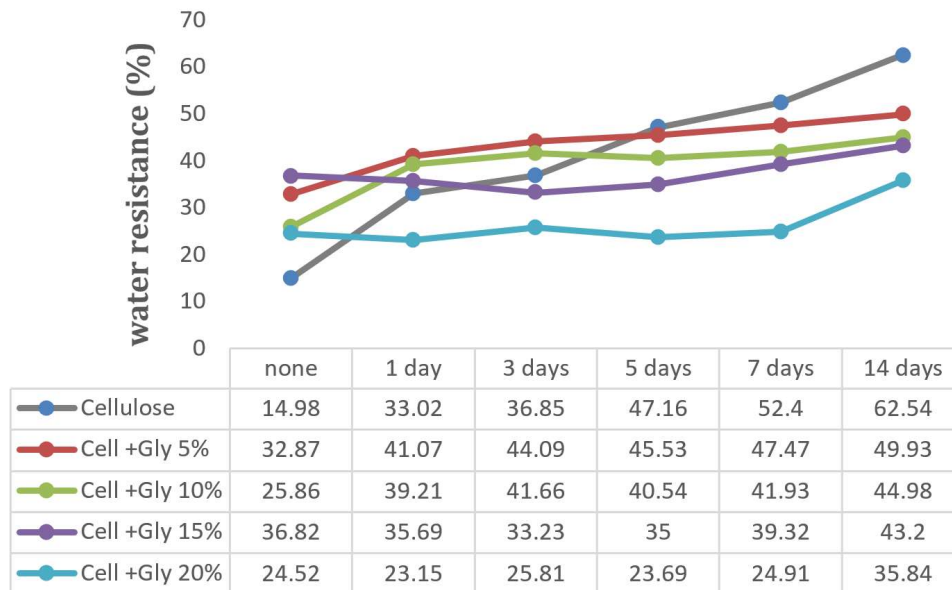
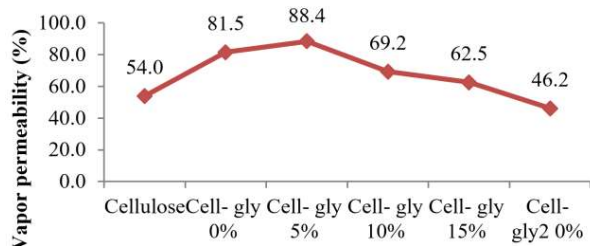


Figure 5. Relationship between water resistance of films



Cellulose- glycerol were immersed for 14 days.

Figure 6. Vapor permeability of Cellulose- Glycerol immerse in lemongrass oils for 14 days

In order to reduce the hydrophilic nature of the film, the lemongrass oil component is a fat with a good protection against moisture. Glycerol addition lessens the film's resistance to steam. This is due to the fact that glycerol can absorb water and is a polar plasticizer with a hydroxyl (OH) group. A lemongrass oil soak results in high water resistance. According to the graph for cellulose film that was exposed to lemongrass oil for 14 days, the more glycerol there is in the film, the less permeable it is to water vapor.

3.3. Functional Groups and Chemical Bonds Analyses

Pure cellulose film's spectra exhibit absorption at the OH group's wave number region, the characteristic peaks of cellulose appeared at 3271.85 cm^{-1} for O-H stretching vibrations at -CH₂OH groups [35], the broader peak for cellulose indicated stronger OH bonding. and in the fingerprint region with a wave number region of 650 cm^{-1} , there was also an aliphatic C-H absorption and peak at 1645 cm^{-1} for C-O stretching vibrations for the glucose carbonyl group [35]. Peak at 1470 cm^{-1} for CH₂ symmetric bending and 1030 cm^{-1} for C-O-C and C-O-H stretching vibrations of the sugar ring [24]. These peaks suggested that BC produced from industrial waste liquid was pure cellulose. Hence the FTIR data reported that the culture medium shows little influence on the functional groups of cellulose samples. Therefore, the synthesized cellulose contained both allomorphs [35].

Figure 7 shows the spectra of cellulose glycerol and spectra cellulose -glycerol after being coated with lemongrass oil. Figure 7a shows the spectra of pure cellulose, 7b shows the FTIR spectra of cellulose-coated citronella oil after soaking for 7 days, then Figure 7c shows the spectra of cellulose with the addition of 20% glycerol, while Figure 7d shows the spectra of cellulose which has been added 20% glycerol and soaked in lemongrass oil for 7 days.

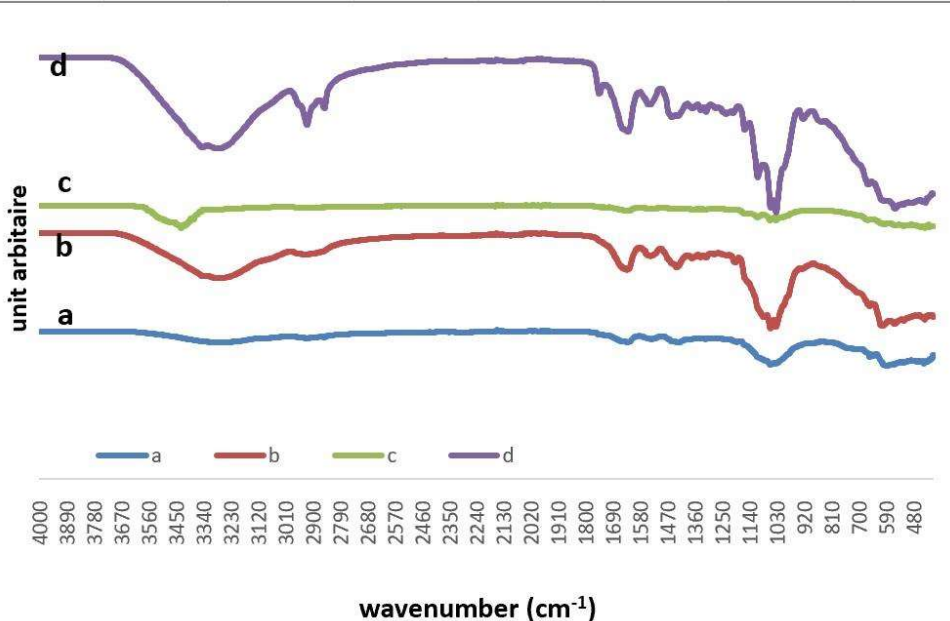


Figure 7. spectra FTIR film a). pure cellulose, b). cellulose-lemongrass oil, c). cellulose- glycerol, d). cellulose-glycerol-lemongrass oil

Addition of glycerol caused a shift in OH absorption from the wave number area of 3271.85 to 3340.33 cm^{-1} [23]. Film of 20% cellulose-glycerol soaked 7 days in lemongrass oil has the sharpest spectra. Glycerol which has more OH groups, causes absorption spectra of OH groups to widen at wave number 3344.90 cm^{-1} [23]. Typical C-H bond stretching from aldehyde uptake is seen by the appearance of two absorbances with medium intensity at wave number 2923.94 cm^{-1} and 2854.09 cm^{-1} [35] which are due to the largest content in lemongrass oil is the citral compound, reinforced by absorption at wave number 1744.01 cm^{-1} which is a group C=O [36].

The clearest spectra were found in the 20% cellulose-glycerol film that was exposed to lemongrass oil for 7 days. Another intense peak at 1054.56 cm^{-1} was assigned to C–O–C pyranose ring stretching vibration [23]. The aromatic C-H stretching group from lemongrass oil absorbs at wave number 3275.56 cm^{-1} , where it combines with the OH group. Since citral compounds make up the majority of lemongrass, two medium-intensity absorptions at wave numbers 2923.94 cm^{-1} and 2854.09 cm^{-1} , which are accompanied by an aldehyde C=O group absorption at wave number 1744.01 cm^{-1} , are indicative of an aldehyde C-H absorption. The cellulose film's OH group is taken up over a wider area due to the 20% glycerol content. The absorption peaks of C=O and C-H aldehydes were sharper and more pronounced after soaking time in lemongrass oil.

4. Conclusions

The glycerol addition alters the material's mechanical characteristics, lowering the tensile strength. The tensile strength, elongation, and Young's modulus of the cellulose film and the cellulose-glycerol film were all increased by the addition of citronella oil. The finest mechanical qualities were provided by 15% cellulose-glycerol films and 5% cellulose-glycerol films that were soaked in citronella oil for 5 days. These films had the highest elongation and the lowest Young's modulus. The film in the C=O group with the highest water and steam resistance and increased absorption intensity was the 5% cellulose-glycerol film that had been exposed to citronella oil for 14 days. Paper that complies with SNI specifications can be substituted with cellulose-glycerol-lemongrass oil for packaging.

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