

# Synthesis of Crosslinked Cellulose/PVA Bioplastic Strengthened with Chitosan as and Alternative to Conventional Plastics

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**Keywords:** Crosslinked cellulose, Chitosan, Conventional Plastics.

**Abstract:** Environment problem from plastic waste should be reduced and solved. The development of biodegradable plastic from cellulose can be a solution to solve it. Cellulose was a natural biopolymer that can be used as bioplastics. Biodegradable plastics made from cellulose can be used as an alternative to conventional plastics, because they are environmentally friendly, easy to obtain and easily degraded. However, used of cellulose-based bioplastic requires physical or chemical modification to improve its physical and mechanical properties. In this study, improvement of physical and mechanical properties of cellulose-based bioplastics was carried out by adding polyvinyl alcohol (PVA), glutaraldehyde crosslinker and chitosan filler. Optimization the synthesis of cellulose / PVA layers was carried out by varying the concentrations of glutaraldehyde and chitosan from 0-56% and 0-33%. The results of the bioplastic film synthesis were evaluated for thickness, swelling and solubility, biodegradability and mechanical properties, the optimum concentration of each variation of glutaraldehyde and chitosan was characterized by FT-IR. The results showed that modification of the cellulose/PVA-crosslinked glutaraldehyde film and the addition of chitosan fillers improve the physical and mechanical properties of bioplastics, with the optimum concentration of each varian being 56% (w/w) and 24% (w/w).

## 1 INTRODUCTION

Plastic is a product that can function as packaging, so that it can facilitate human work. The high use of plastic packaging causes an accumulation of plastic waste, which causes environmental pollution (Selvamurugan & Sivakumar, 2019). Plastic packaging, especially food packaging that is produced non-biodegradable, leads to environmental problems because it takes thousands of years to degrade. Biodegradable means that these materials can be completely consumed by microorganisms without leaving harmful pollutants in the environment. To solve this problem, one solution is to replace the non-biodegradable plastic base material into a easily degraded material, namely biodegradable plastic (Pikon & Czop, 2014). The material used for the manufacture of plastics plays an important role in determining the quality of the films produced. In the manufacture of films can mix natural polymer materials and synthetic polymer materials (Isroi & Cifriadi, 2017).

In recent years, the use of natural fibers as reinforcements in polymers has attracted much

attention due to the environmental concern. Cellulose fibers exhibit high mechanical properties. Besides, they also offer a number of other advantages over conventional reinforcing materials, such as low cost, worldwide availability, and biodegradability. Particular attraction is its low density which leads to high specific mechanical properties (Niu, et al. 2015). These unique properties have made cellulose an ideal candidate for high performance biocomposites. Various biodegradable polymers, such as chitosan (Angadi, et al. 2012), starch, polycaprolactone (PCL) (H.Lonnberg, et al. 2011), poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) (E. Ten, et al. 2010), polylactic acid (PLA) (L. Suryanegara, et al. 2010), poly (furfuryl alcohol)(PFA) (H. Dekaa, et al. 2013), and polyvinyl alcohol (PVA) (Priya, et al. 2014) have been explored as potential matrices for this kind of composites. PVA is the most widely produced watersoluble synthetic polymer in the world. It is also a versatile polymer with broad applications due to its biodegradability, biocompatibility, high tensile strength, excellent adhesive properties, chemical resistance and gas barrier properties (Niu, et al. 2015).

Plasticizers play a role in increasing the flexibility and permeability of cellulose to water and gas vapor. Synthetic polymer which is biodegradable as a mixture of cellulose for the manufacture of biodegradable plastic films is PVA. PVA is one of the largest water-soluble, non-toxic and environmentally friendly polymers. PVA has a polar hydroxyl group attached to the carbon which makes it very hydrophilic. In addition, PVA is widely used in the plastic packaging industry because of its solubility (Abdulkani, et al. 2013). Although both cellulose and PVA have plenty of hydroxyl groups along their molecular chains, direct incorporation of pristine cellulose fibers into PVA matrix cannot produce composites with good mechanical properties. This is due to the fact that most hydroxyl groups in cellulose and PVA molecules have already formed either intra- or inter-molecular hydrogen bonds. Therefore, it is difficult to form new hydrogen bonds between the two components by simple mixing (Niu, et al., 2015).

Chemical crosslinking in composites is an efficient way to achieve desired compatibility [4]. Chabba et al. crosslinked soy flour/flax yarns composites using glutaraldehyde (GA) as the crosslinking agent to improve the tensile and thermal properties (Chabba, et al., 2015). Li et al. also used GA to prepare crosslinked chitosan/PVA blend beads with high mechanical strength (Li, et al., 2007). Glutaraldehyde is a crosslinking agent that has the ability to bind hydroxyl groups to polymeric materials. Glutaraldehyde is also one of the crosslinking agents which can cause the matrix crosslinking to be tighter and increase the tensile strength. In addition, it can improve the mechanical properties of the mixed film and can improve the characteristics of water resistance and film thermal stability (Basuki & Sanjaya, 2009).

To increase the strength and hardness of bioplastic products, it can be increased by adding fillers. Chitosan is a polysaccharide that is abundant in nature after cellulose. The use of chitosan as an additive in making plastic films serves to improve the transparency of the plastic film produced. The more chitosan is used, the better the mechanical properties and air resistance of the bioplastic products. In addition, chitosan is non-toxic, easily biodegradable and is polyelectrolytic (Mollah, et al., 2016).

Therefore, this research will conduct the development of bioplastic films that are innovative, environmentally friendly and biodegradable. The process of making the film was carried out by preparing the bioplastic film Cellulose / PVA with

added glutaraldehyde as cross-linker with the addition of chitosan filler as reinforcement.

## 2 RESEARCH METHODS

### 2.1 Materials

Commercial cellulose (99%-cellulose). PVA was purchased from Merck Company (Germany). glutaraldehyde, chitosan, dimethyl sulfoxide (DMSO) was purchased from Merck MERCKMILLIPORE (Germany),  $\text{CH}_3\text{COOH}$ , distilled water, filter paper from the Universitas Indonesia Materials Technology Laboratory.

### 2.2 Cellulose Activation

Cellulose (0.5 g) was immersed in solvent (water: DMSO :: 1:3) for 24 hours to swell and to activate reactive sites on the surface (Kohli, et al., 2017).

### 2.3 Bioplastic Film Synthesis

#### 2.3.1 Cellulose / PVA Crosslinked Glutaraldehyde Film

The synthesis process begins with the manufacture of cellulose / PVA. A total of 2 g of PVA was dissolved in 23 mL of distilled water, heated at 80°C for 1 hour while stirring at high speed. Then add cellulose which has been activated overnight and stirred again for 30 minutes at high speed. Then added glutaraldehyde (0-56%, w/w). Then the bioplastic is molded and left at room temperature overnight. Then, oven for 5 hours at a temperature of 60°C.

#### 2.3.2 Cellulose / PVA Filler Chitosan Film

Solution of 1% chitosan (w/w) was made using 2% (w/w) acetic acid solution. The chitosan solution (0-33% w/w) is added to the cellulose / PVA / glutaraldehyde mixture by the process described in 2.3.1. The mixing is carried out right after the crosslinking agent is added, before the mixture thickens and becomes agar. Then the bioplastic is molded and left at room temperature overnight. Then, oven for 5 hours at a temperature of 60°C.

## 2.4 Film Characterization

### 2.4.1 Measurement of Film Thickness

The thickness of the samples was determined with a couplers micrometer. This tool has an accuracy of up to 0.01 mm. Measurements were repeated in ten different regions of each sample. Average values were calculated and used (Abdollahi, et al., 2012).

### 2.4.2 Swelling and Water Solubility Measurements

The sample was cut into small pieces (2x2 cm) and weighed to determine their dry mass. The weighed samples were placed in closed beakers containing 30 mL of water and stored at temperature room. The kinetic of swelling was evaluated by periodically measuring the weight increment of the samples. The film is taken, cleaned and shaken several times to remove the liquid that is on the surface of the bioplastic film and weighed. The weighing was continued until equilibrium state. The procedure was repeated three times for each sample to confirm its repeatability. The water gain of each sample was calculated as follows:

$$\text{Swelling (\%)} = \frac{W_s - W_0}{W_0} \times 100\%$$

where  $W_s$  is the weight of the film sampel after immersion and  $W_0$  is the weight of the initial film sampel (Abdollahi, et al., 2012).

After that, the film from the swelling test is dried until the mass of the sample film is constant and the solubility is calculated using the equation:

$$\text{WS (\%)} = \frac{W_k - W_0}{W_0} \times 100\%$$

Where  $W_k$  is weight of the dried layer after swelling test (Sa'adah, 2020).

### 2.4.3 Biodegradability in Soil

In this method, samples films 20 mm × 20 mm × 1 mm small pieces were weighted and placed for 120 days into the agricultural soil in a pot. The pot was covered with a plastic net and exposed to atmospheric conditions for 120 days. Variations in film morphology, the time of films disintegrated and weight loss were recorded. To determine the weight loss the specimen of each sample was quickly washed with cold water and dried in an oven at 70 °C to constant weight. The weights of the sample, before and after washing were recorded (Priya, et al. 2014).

### 2.4.4 Mechanical Properties

Tensile strength (TS) and elongation at break (E%) of the film samples were determined according to ASTM standard method D882–02 with an Strograph EII Testing Machine. The film samples were cut in rectangular specimens (40x20 mm). The test is done by clamping both ends of the sample to a tensile testing machine. Then the start knob is turned on and the tool will pull the sample until it breaks. Saved and recorded the results of tensile strength and% elongation that are on the tools.

### 2.4.5 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectra were studied with a Nicolet 560 spectrometer (Nicolet Co., USA). KBr solids are smoothed and inserted into the holder for background measurement. Bioplastic film samples on top of the holder and measured in the wave number range 500-4000  $\text{cm}^{-1}$  (Abdollahi, et al., 2012).

## 3 RESULTS AND DISCUSSION

### 3.1 Film Thickness

Bioplastic film thickness measured using a micrometer. Thickness is one of the parameters used to determine the quality of a bioplastic characteristic. The results showed that the thickness of the bioplastic did not increase significantly with an increase in the glutaraldehyde crosslinker concentration. However, at certain concentrations it will increase the thickness of the printed plastic (Table 3.1). The results showed that all variations of glutaraldehyde and chitosan had the same film thickness. The same thickness value caused because the dissolved material evaporates when heated.

Table 1 Film Thickness

Treat ment	Glutaraldehyde (%w/w)	Thick ness (mm)	Treat ment	Chitosan (%w/b)	Thickness (mm)
PPP <sub>0</sub>	0	0,12	PPP <sub>0</sub>	0	0,12
PPP <sub>30</sub>	30	0,20	PPP <sub>12</sub>	12	0,20
PPP <sub>46</sub>	46	0,20	PPP <sub>24</sub>	24	0,20
PPP <sub>56</sub>	56	0,20	PPP <sub>33</sub>	33	0,20

Bioplastic thickness influenced by the area of the mold, the volume of the solution and the total amount of dissolved solids (Sa'adah, 2020). In this study, the mold area was made the same, so the influencing factor was the volume of solute glutaraldehyde and chitosan. So, the addition of glutaraldehyde and chitosan increases the thickness.

### 3.2 Swelling and Water Solubility

The swelling test was conducted to determine the interaction between the bioplastic film and water. Conventional plastics circulating in the community are hydrophobic, making it difficult to dissolve in water. Therefore, the development of bioplastic films which are difficult to dissolve in water is carried out (Sa'adah, 2020).

The results of the swelling and solubility test are shown in Figures 1 & 2. Based on the results of the study, the higher the concentration of glutaraldehyde and chitosan added, the lower the % increase in mass or swelling of bioplastics. This is due to the hydrogen bonding that occurs between glutaraldehyde and cellulose as well as chitosan and cellulose in the bioplastic film, which prevents the formation of holes that can be traversed by water. In general, the lower the swelling ability of the bioplastic film, the lower the film solubility. This is because the lower the amount of water that enters the cavity, the less the film component can dissolve (Niu, et al. 2015, Abdhulkani, et al. 2013, Basuki & Sanjaya, 2009).

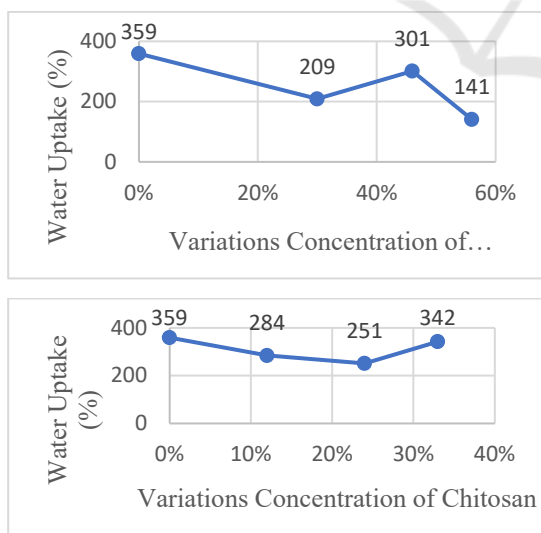


Figure. 1 Swelling

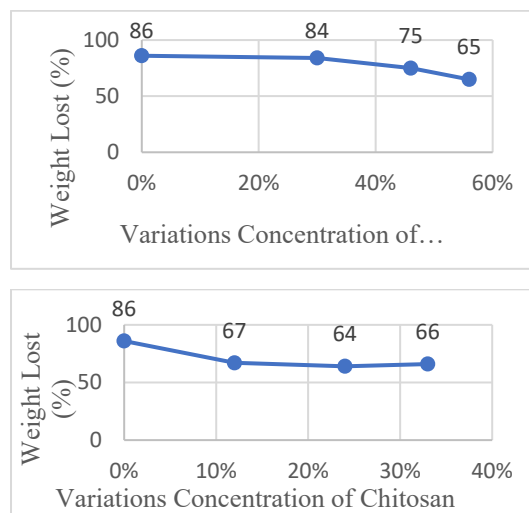


Figure 2. Water Solubility

The results showed that in general the swelling ability of bioplastics decreased with increasing concentrations of glutaraldehyde and chitosan (Figure 1). The results showed that the % variation in the development of glutaraldehyde and the lowest chitosan in this study was 24% PPP56 and PPP24, respectively, with a percentage of 141% and 251%. Meanwhile, the highest % swelling was the PPP0 variation with the proportion of 359%. Glutaraldehyde acts as a crosslinker and also acts as a plasticizer. The PPP56 variation has higher intermolecular interactions between molecules compared to other variations, so that in other variations the ability of glutaraldehyde to act as a plasticizer. The results showed that the higher the concentration of chitosan was added, the lower the percentage increase in mass or swelling of bioplastics. This is due to the hydrogen bonding that occurs between chitosan and cellulose on the bioplastic film, so that it prevents the formation of cavities that can be passed by water (Sa'adah, 2020).

The solubility test results were obtained from the dried swelling bioplastic film, then the final mass of the sample was calculated. There is a correlation between the results of the swelling test and the results of the solubility test. The higher the swelling ability of the bioplastic film, the more dissolved bioplastic film mass will be shown (Figure 2). Addition of glutaraldehyde and chitosan in Cellulose / PVA matrix can reduce the solubility of films in water. The highest yield of bioplastic film solubility at the PPP0 variation was 86%. For the glutaraldehyde variation, the lowest solubility yield was 65% for PPP56. For

chitosan variation, the lowest solubility yield was 64% for PPP24. The results obtained are linear with the results of the swelling test. This will increase the interaction between molecules and reduce sensitivity to water (Basuki & Sanjaya, 2009).

### 3.3 Biodegradability in Soil

Bioplastic film biodegradation testing was carried out by means of the burial test method in the soil. The biodegradation test aims to determine the level of resistance of the bioplastic film in the soil for a certain time. The results of the biodegradation analysis showed that all variations experienced a decrease in the mass of the bioplastic film, the mass decrease for each variation did not experience any difference significant (Figure 3 and 4). The decrease in the mass of the bioplastic film is an indication that the bioplastic film is biodegradable. The reduction in the mass of the bioplastic film was caused by the attack of microorganisms and the absorption of water content (swelling) of the bioplastic film which is in the soil, initiates a hydrolysis reaction so that the bioplastic polymer can be decomposed into smaller compounds (Sa'adah, 2020).

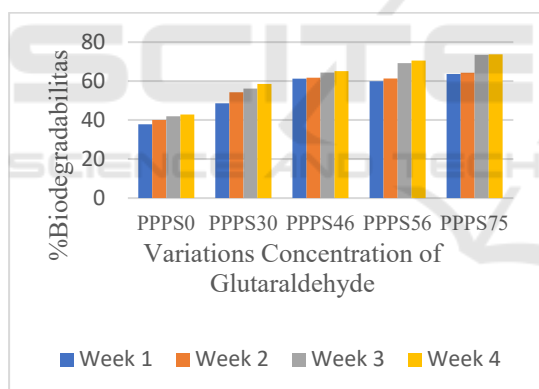


Figure. 3 Biodegradability Variation of Glutaraldehyde

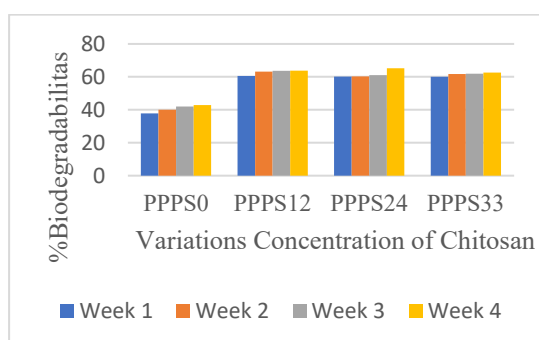


Figure. 4 Biodegradability Variation of Chitosan

The results showed that the higher the concentration of glutaraldehyde and chitosan was added, the % biodegradability of bioplastics increased. The results showed that the higher the concentration of glutaraldehyde added, the % biodegradability of bioplastics was increased. Glutaraldehyde acts as a crosslinker and plasticizer, where the increased glutaraldehyde concentration will increase its ability as a plasticizer. This can reduce the interaction between macromolecules, so that the bonds between macromolecules are easier to break, so that the bioplastic film will be more easily degraded by microorganisms. In addition, the hydrophilic groups present in glutaraldehyde will make the degradation process easier, thus making bioplastics more environmentally friendly (Basuki & Sanjaya, 2009).

The increase in the amount of chitosan in bioplastics causes the amount of carbon to also increase. Carbon is one of the elements needed by microorganisms as a food source, increasing the amount of chitosan will increase the carbon source that can be used as food for microorganisms in the soil. The % increase in biodegradability is due to increased hydrolysis of random chain cutting which triggers a reduction in molecular weight. The reduction in molecular weight will trigger these molecules to become smaller molecules, so that they will be easier to degrade by microorganisms (Abdullah, et al., 2020). There are various kinds of microorganisms that have been isolated from the soil using bioplastics as a carbon source. Some of these microorganisms include Actinobacteria species such as Amycolatopsis, Thermomactimuces, Actinomadura, Nanomuraca, Laceyella and Streptomyces, of which the most common species are Amycolatopsis and Streptomyces. In addition, there are also species of Paenibacillus, Pseudomonas, Bacillus and Bulkholderia. Several types of fungal species that have been isolated and are responsible for degrading bioplastics are Aspergillus, Fusarium and Penicillium, Penicillium is the most common species (Emadian, et al., 2017).

### 3.4 Mechanical Properties

The mechanical strength test of bioplastics is carried out by measuring the tensile strength and strain (% elongation). Tensile strength (tensile strength) is the ability of a bioplastic film to withstand a given load until the bioplastic breaks, while % elongation is the optimum stretch extension or increase in bioplastic when it is pulled to break. % elongation is used to measure the elasticity of a polymer.



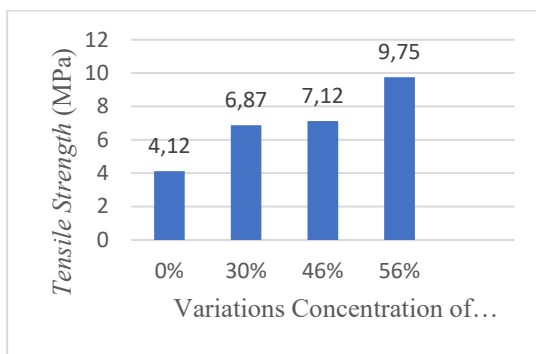


Figure 5. Tensile Strength

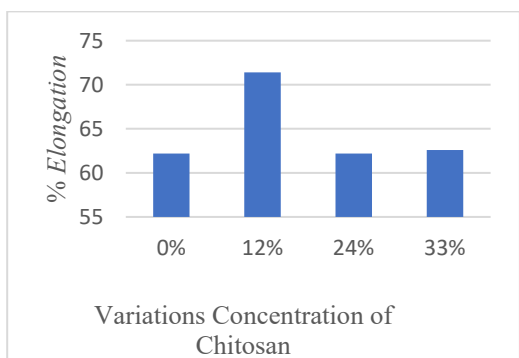
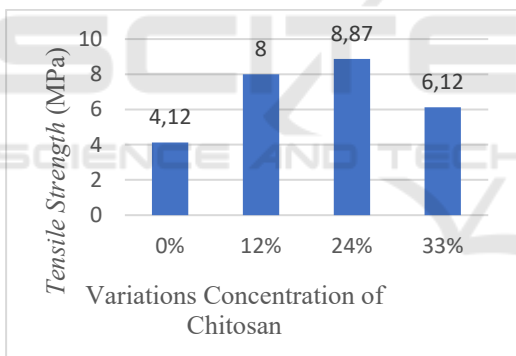
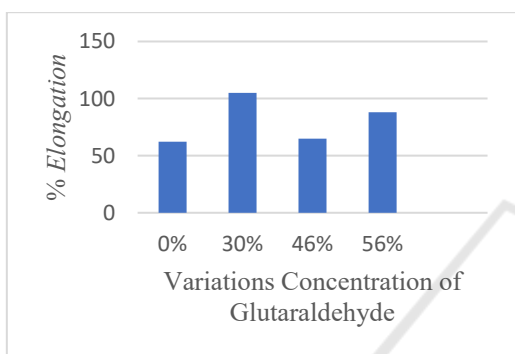


Figure 6. Elongation

The presence of cross-linking will also increase the molecular weight of the bioplastic, where the tensile strength of the polymer increases with increasing molecular weight and reaches a saturation level at a certain molecular weight. At lower molecular weights, the polymer chains are loosely bound and the chains are easier to move so that the strength of the polymer is low (even though it has a higher crystallinity), whereas polymers with large molecular weights have large chains and give strength to the polymer. The results of the bioplastic tensile strength test with variations in the concentration of chitosan are shown in Figure 5. The results of the tensile strength test showed that the higher the concentration of chitosan added, the tensile strength of the bioplastic was increased. This is due to the presence of filler so that it can improve the mechanical properties of the bioplastic film. The improved mechanical properties of bioplastics can also be attributed to the good interface adhesion, which can form hydrogen bonds between cellulose and filler chitosan (Sa'adah, 2020).

The effect of glutaraldehyde on% elongation is shown in Figure 6. The parameter of% elongation is used to determine the flexibility and tensile strength of the bioplastic film. An increase in the glutaraldehyde concentration will increase the% elongation of the bioplastic film. This is because the role of glutaraldehyde as a plasticizer can reduce the interaction of the intermolecular bonds of the bioplastic polymers and replace them with hydrogen bonds formed between the plasticizer and the polymer, thereby reducing stiffness and increasing the flexibility of the film. The addition of chitosan to bioplastics will reduce the% elongation (elongation) of the bioplastic. This is because the addition of chitosan will increase the stiffness of the bioplastic, thus reducing the% elongation ability of a bioplastic film. In addition, increasing the amount of chitosan filler will increase the hydrogen bonding interaction between the filler and the cellulose matrix, thereby reducing the flexibility of the bioplastic film (Mollah, et al., 2016).

### 3.5 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The analysis of functional groups on the effect of adding glutaraldehyde and chitosan was carried out by FTIR, which is shown in Figure 7. In the FTIR spectra of cellulose / PVA / glutaraldehyde films showed a widened peak at wave number 3200-3500  $\text{cm}^{-1}$  indicating the presence of an OH group. The absorption band at the numbers 1700-1715  $\text{cm}^{-1}$

shows the peak area for the aldehyde group. The FTIR spectra results of the cellulose / PVA / glutaraldehyde / chitosan film showed absorption at the wave number  $3445.1 \text{ cm}^{-1}$  which is the  $-\text{OH}$  region and water. The absorption band at wave number  $2928 \text{ cm}^{-1}$  shows the peak area for the C-H group. The absorption at wave numbers  $1659.7$  and  $1376.1 \text{ cm}^{-1}$  is the peak area for the N-H group of the amine group and the C-H group on  $\text{CH}_3$  (Basuki & Sanjaya, 2009).

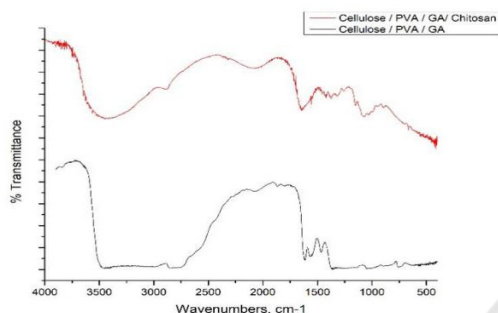


Figure 7: FTIR analysis

Figure 7. Results of the cellulose/PVA/glutaraldehyde/chitosan

## 4 CONCLUSION

Based on the research that has been done, it can be concluded that the variation in the concentration of cross-linking agent glutaraldehyde and chitosan filler has an effect on the physical and mechanical properties of the bioplastic film layer, with each optimum concentration to improve the physical and mechanical properties of the bioplastic is 56% (w/w) and 24% (w/w). Evidenced by the % swelling and the lowest solubility and the highest tensile strength values. Increasing the concentration of glutaraldehyde and chitosan at a certain concentration decreased the physical and mechanical properties of the bioplastic film.

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