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## SYNTHESIS AND CHARACTERIZATION OF BIOPLASTICS FROM CORN-STALK FIBER AND CHITOSAN

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### ABSTRACT

The plastics used by the public are generally made of petrochemicals which take a long time to degrade completely. One of the solutions to overcome the problem of plastic waste is to make produce bioplastics. Bioplastics are made from renewable natural materials and are environmentally friendly. One of the materials that can be made into bioplastic is chitosan. Chitosan has been widely used in the packaging industry, especially in food packaging, because of its biocompatibility, biodegradability, and non-toxicity. However, the relatively high price of chitosan will limit the use of chitosan film in various applications. In this study, bioplastics were made from chitosan and cellulose. Cellulose was obtained from the corn-stalks waste extract. The results showed that the glycerol addition increases the swelling degree, decreases the tensile strength, increases the elongation break, and accelerates the degradation of bioplastics. The optimum conditions for producing bioplastics were obtained by 1% glycerol addition, cellulose: chitosan ratio of 25:75, and 1% glutaraldehyde addition.

**KEYWORDS:** Bioplastics, Chitosan, Cellulose, Corn-stalk

### 1. INTRODUCTION

The production of plastic materials in Indonesia reached 7.23 million tons in 2018, with a growth rate of about 5% per year. In 2019, plastic waste reached 9.52 million tons [18]. Plastics circulating in the community are generally made from petrochemicals that have a prolonged biodegradation rate and take a

long time to degrade completely. If this is allowed to continue, this will cause environmental pollution problems. Usually, handling plastic waste is only disposal, landfill, burning, or recycling. Carbon emissions from combustion can be a new source of environmental pollution [1].

One of the solutions to overcome the problem of plastic waste is to make bioplastics. Bioplastics are made from renewable natural materials and have environmentally friendly properties. Chitosan is a natural polysaccharide that can be obtained from the deacetylation of chitin in the shells of invertebrate animals. Chitosan is widely used in the packaging industry, especially in food packaging, due to its biocompatibility, biodegradability, and non-toxicity. Chitosan has a cationic (amine) group and an anionic (hydroxyl) group resulting in a good film with high strength [19]. In addition, chitosan also has a hydrophobic acetyl group that makes bioplastics more resistant to water. However, the price of chitosan is relatively high. This challenge will limit the use of chitosan film in various applications [15]. Therefore, additional materials are needed to produce chitosan bioplastics.

Cellulose is a natural polymer with biodegradable properties and can decompose up to 67% within 2-3 weeks [5]. Cellulose is found in many agricultural wastes, including corn stalk waste [25]. Corn stalks have renewable properties, biodegradability, and abundant availability [26]. Corn stems contain 42.6% cellulose, 21.3% hemicellulose, and 8.2% lignin. With the high content of cellulose, which is more than 40%, corn stalks can potentially become bioplastic raw materials.

The research aims to make bioplastics based on chitosan and cellulose from corn stalks. Corn stalks were chosen because they contain large amounts of cellulose. The effect of glycerol addition and crosslinking agent of glutaraldehyde on the characteristics of bioplastic products was investigated.

## 2. MATERIALS DAN METHODS

### 2.1 Materials

Corn-stalk was obtained from Kebumen, Indonesia. Chitosan (deacetylation degree of 85.78%) was purchased from Biotech Surindo, Cirebon, Indonesia. Sodium hydroxide (NaOH), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), acetic acid (CH<sub>3</sub>COOH), glycerol, and glutaraldehyde were purchased from Merck, Germany. Aquadest was produced by a homemade integrated membrane unit (conductivity 1.7 μΩ/cm).

### 2.2 Pretreatment of Corn-stalk fiber

Corn stalks were cut into ±5 cm and dried in direct sunlight. The dried corn stalks were crushed with a blender, sieved using a 60-mesh sieve, and then dried again. The crushed corn stalks were suspended in NaOH 25% (w/v) solution at 100°C for 1 hour. The residues were collected and washed extensively with

water until pH neutral and dried at 65°C. The results showed that the cellulose content of corn stalks was 47.706%.

### 2.3 Bioplastics synthesis

Bioplastic synthesis was done by mixing corn stalk fiber with chitosan with various mass ratios (w/w). The mixture was dissolved in 1% acetic acid solution at a concentration of 3% (w/v). The mixture solution was heated at a constant temperature of 70°C and stirred at 700 rpm for 30 minutes. Glutaraldehyde was then added to the mixture and stirred for 60 minutes. The mixed solution was left for 24 hours to remove air bubbles. Subsequently, the solution was poured into the mold and soaked in 2% NaOH solution at room temperature for 24 hours. The bioplastic was removed from the mold and dried in ambient conditions.

### 2.4 Characterization of Bioplastic

- Swelling degree

The dried bioplastic was weighed and then immersed in water for 8 hours. The wet bioplastic was carefully wiped with a tissue to remove excess water and then weighed again. The swelling degree was determined by Eq. (1)

$$SD = \frac{w_2 - w_1}{w_1} \times 100\% \quad (1)$$

where  $w_1$  and  $w_2$  are the bioplastic weight before and after water immersion

- Mechanical properties

In order to evaluate the mechanical properties of the bioplastic, tensile strength and elongation at break of the bioplastics were determined. The tests were carried out with the Ta Plus Texture Analyzer.

- Biodegradation test

The dried bioplastic was weighed and then buried in the ground for seven days. After the periods of stockpiling, the weight of decomposed bioplastic was measured. The biodegradability of bioplastic was determined by calculating the difference between weight before and after being buried and then divided by initial weight [11].

- Functional groups

The primary functional groups of the bioplastics were investigated using Fourier Transform Infrared (FTIR) (Shimadzu IR Prestige-21). In this analysis, the spectra of bioplastics have been compared.

## 3 RESULT AND DISCUSSION

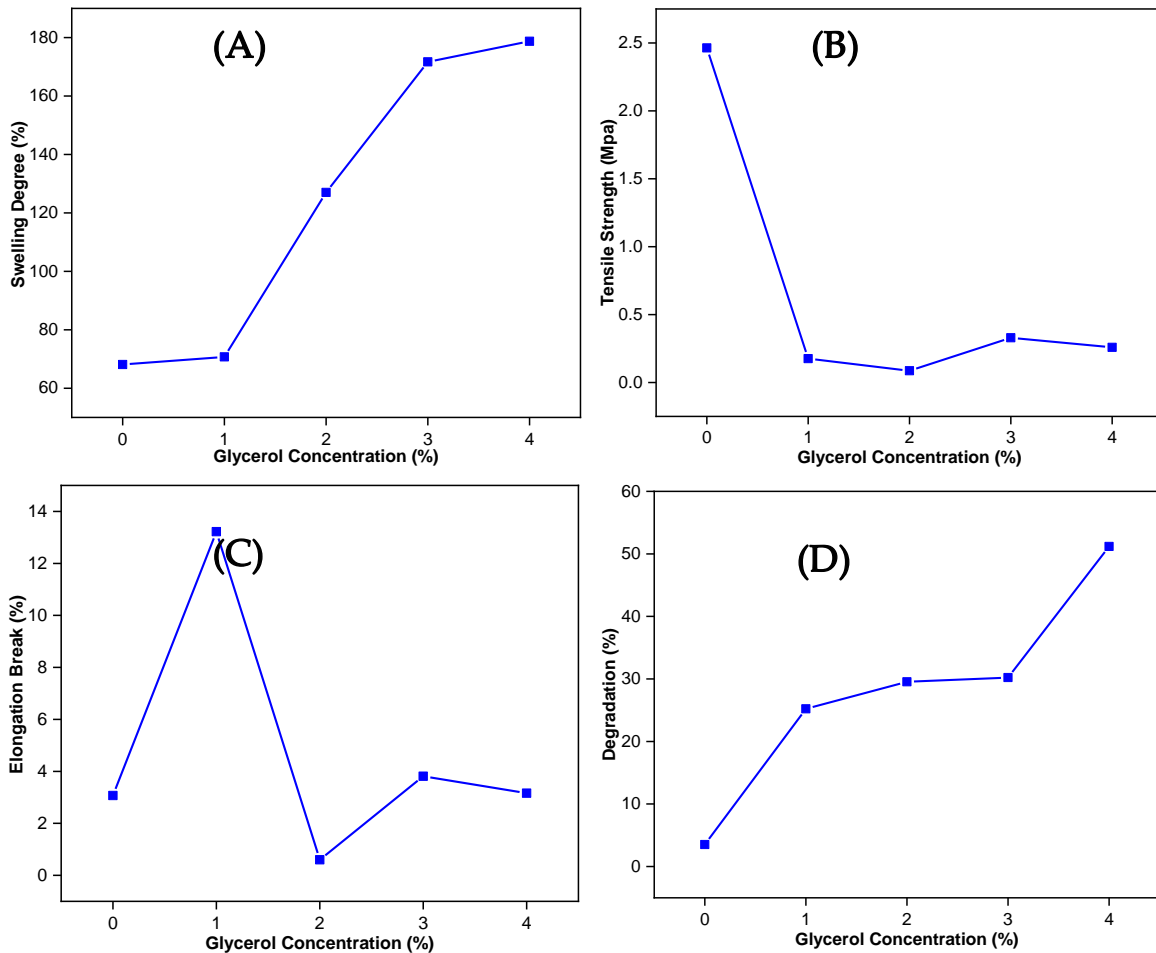
### 3.1 Effect of Glycerol Addition on Bioplastic Characteristics

Biodegradable films made from cellulose fibers are suitable. Additional plasticizers should be introduced to increase the film's elasticity. Plasticizers are commonly used to enhance the flexibility and stretchability of cellulose substrates [14, 24]. In this study, glycerol was used as a plasticizer.

The swelling degree was evaluated by measuring the change in the weight of the bioplastic in the aquadest. The results are shown in Fig 1(A). The addition of glycerol can increase the swelling degree of the bioplastic. These results imply that adding glycerol enhances the bioplastics hydrophilicity. Glycerol has three hydroxyls, which are hydrophilic and hygroscopic; therefore, it easily absorbs water [27]. Bioplastics are said to be good when they have water-resistant properties. The decreasing swelling degree value indicates that bioplastics have lower heritability (ability to absorb water). Therefore, a low swelling degree of bioplastics is desirable [32].

The tensile strength test aims to determine the maximum stress a material can withstand before breaking. From Fig. 1(B), it can be seen that with the increase in the glycerol concentration added, the bioplastic's tensile strength value decreases. Increasing the glycerol concentration leads to a decrease in tensile strength because it reduces intermolecular interactions by allowing more shear chains and increasing the elongation of the bioplastic. Increasing glycerol concentration resulted in a significant reduction in tensile strength [4]. The hygroscopic nature of glycerol contributes more to the plasticizing effect than other plasticizers, thus increasing the mobility of the polymer chains and increasing the flexibility of bioplastics [9].

The elongation test determines the maximum change in bioplastic length before breaking (plastic elasticity). The elongation test was carried out by comparing the increase in length before and after the tensile test was taken. The effect of the glycerol addition on the elongation break value presented in Fig. 1(C). The addition of glycerol at low concentrations can increase elongation at breaks. However, the elongation at break value decreases at a concentration of glycerol addition above 1%. The glycerol addition can break hydrogen bonds in adjacent polymer molecules, increasing the intermolecular distance [28]. As a result, the stiffness of the polymer molecular chains will decrease, and the flexibility of bioplastics will increase [16]. However, if the addition of the glycerol concentration exceeds the saturation value, the glycerol will be in its phase outside the cellulose-chitosan phase. This condition causes the elongation break value to decrease [6].



**Figure 1. Effect of glycerol addition on bioplastic characteristics: swelling degree (A), tensile strength (B), elongation break (C), degradation (D)**

The primary raw material of bioplastics is biopolymer. The hydroxyl groups in biopolymers, which are hydrophilic, cause water to be easily absorbed by bioplastics and then microorganisms in the environment to quickly enter the plastic matrix. The addition of glycerol which is hydrophilic, can increase the free volume between the cellulose polymer chains and increase the free hydroxyl groups that will form hydrogen bonds with the liquid. Thus, increasing glycerol addition increased the ability of bioplastics to absorb liquids and increased the biodegradability of bioplastic (Fig. 1(D)) [2].

According to SNI 7188.7:2016, the elongation at break of biodegradable plastic is 10-20% [22]. In this study, the elongation at break of bioplastics by adding 1% glycerol was 13.2%. Besides that, based on the

international plastic standard (ASTM5336), the overall biodegradability for PLA (Polylactic Acid) plastic from Japan and PCL (Polycaprolactone) from England takes 60 days [23]. In this study, the percentage of bioplastic degradation by adding 1% glycerol for seven days reached 25%. Therefore, the optimum addition of glycerol to bioplastics is 1%.

### 3.2 Effect of Cellulose/Chitosan Ratio on Bioplastic Characteristics

To improve the characteristics of bioplastics, the study of the effect of the cellulose/chitosan ratio on bioplastic products has been carried out. Fig. 2 shows that decreasing the cellulose/chitosan ratio causes the increase swelling degree of the resulting bioplastic. On the other hand, the decrease of cellulose/chitosan ratio reduce, the tensile strength and degradation of bioplastics. The hydrophilicity of chitosan is higher than cellulose, so the rise of chitosan content in bioplastics enhances the swelling degree and reduces the tensile strength [13, 15].

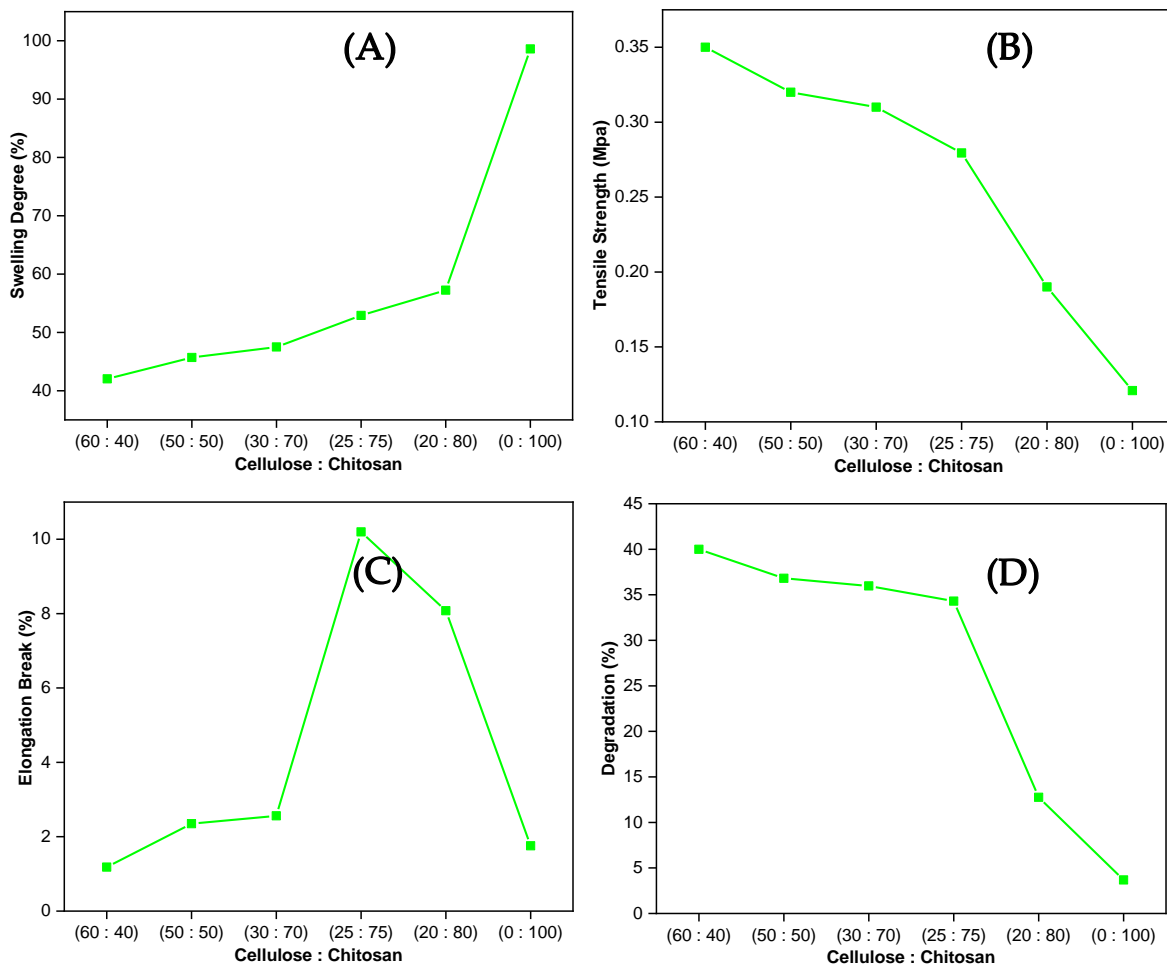


Figure 2. Effect of cellulose: chitosan ratio on bioplastic characteristic: swelling degree (A), tensile

**strength (B), elongation break (C), degradation (D)**

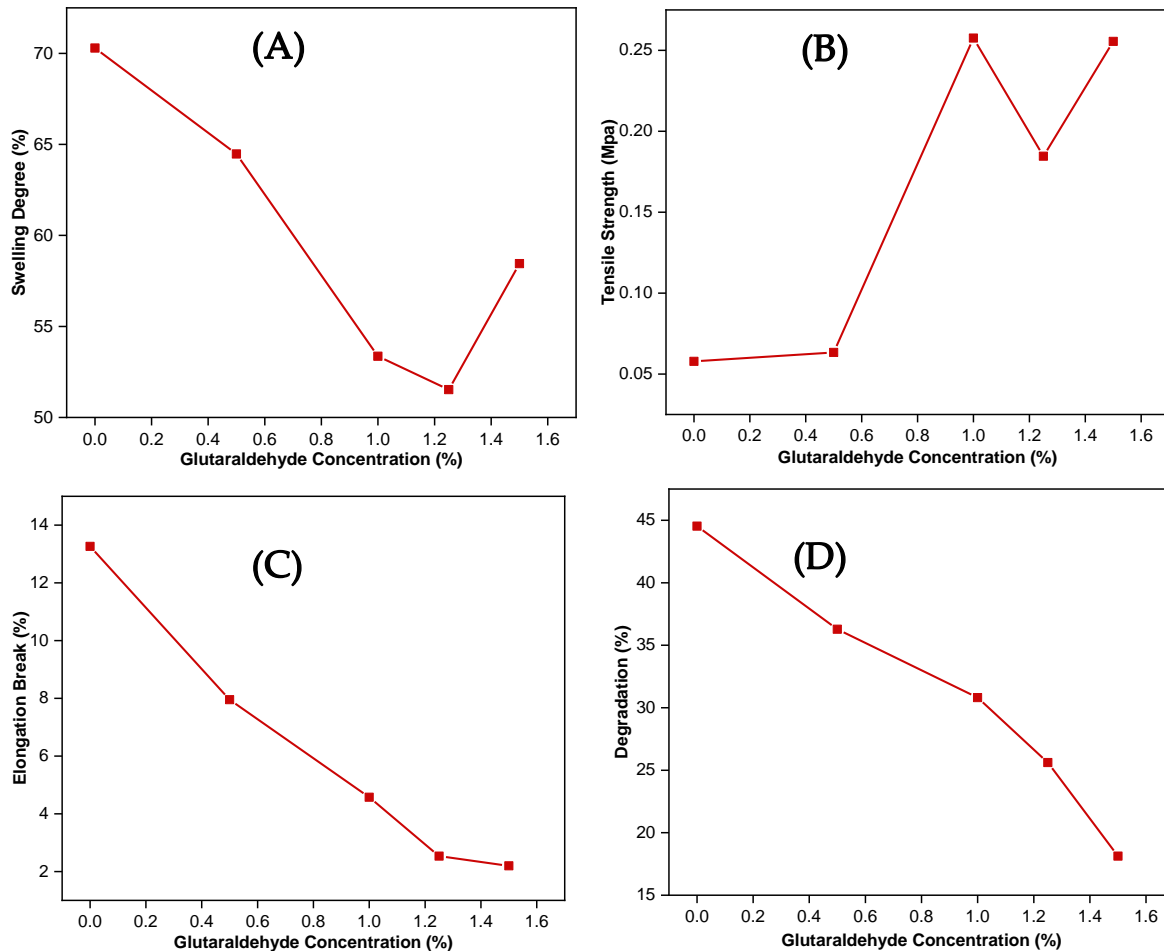
The decrease in the cellulose/chitosan ratio up to 25:75 (w/w) increases the elongation break value. However, the elongation at break value declines at the lower cellulose/chitosan ratio. The amine group ( $\text{NH}_2$ ) in the chitosan compound, which is protonated to  $\text{NH}_3^+$ , will form hydrogen bonds with the hydroxyl group ( $\text{OH}^-$ ) in cellulose. These hydrogen bonds create an interactive network between polymer chains, making the material more elastic. The higher elongation at break value indicates that bioplastics are more flexible. [7].

The chitosan excess causes a reduction in the distance of intermolecular bonds because there are no free OH groups in cellulose that bind to groups on chitosan. This causes the availability of movement at the bioplastic interface to be low so that it becomes more rigid and less elastic [29].

Fig. 2(A) also shows that decreasing the cellulose/chitosan ratio decreases the biodegradability of bioplastics, and the percentage value of degradation decreases significantly when the raw material of bioplastic approaches pure chitosan. Hydrophilic polymer bioplastics exhibit rapid biodegradation [10]. The reduced weight of bioplastics during soil burial shows the natural environment's degradation due to the activity of microorganisms [11]. Chitosan has antimicrobial properties that can prevent microbial attack. Thus, more chitosan content in bioplastics leads to reduced weight loss than in bioplastics with lower chitosan concentrations [3].

**3.3 Effect of Glutaraldehyde Crosslinking on Bioplastic Characteristics.**

Fig. 3(A) shows that the swelling degree decrease with the increase of glutaraldehyde concentration. Crosslinking cause interconnections between chitosan polymer chains and reduce the mobility of the material, thereby reducing the swelling degree [17]. Glutaraldehyde increases the crosslinking density of the polymer chains. When the concentration of glutaraldehyde increases, the hydroxyl and amino groups, which have hydrophilic properties, are crosslinked to weaken the sample's heritability (ability to absorb water) [32].



**Figure 3. Effect of glutaraldehyde crosslink on bioplastic characteristic: swelling degree (A), tensile strength (B), elongation break (C), degradation (D)**

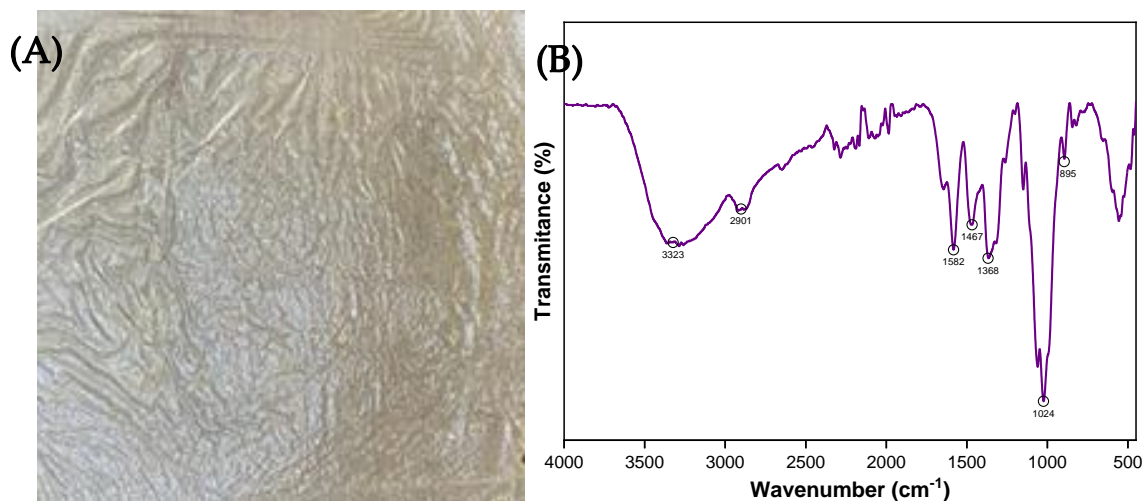
The rise of glutaraldehyde concentration increases the tensile strength value of the bioplastic (Fig. 3(B)). Tensile strength shows the resistance of a material to break. The addition of glutaraldehyde causes the formation of crosslinks between the amine group and the hydroxyl group with the aldehyde group in glutaraldehyde. The crosslinking in chitosan are highly reactive, particularly between the aldehyde and free amino groups. When incorporating glutaraldehyde above a concentration of 2%, the tensile strength value tends to be constant because no free amine and hydroxyl groups could bind to glutaraldehyde [1]. The increasing glutaraldehyde concentration reduces the elongation at break of bioplastic (Fig. 3(C)). The formation of crosslinked networks between amine groups and hydroxyl groups with aldehyde groups in glutaraldehyde causes the matrix to become more compact and stiffer, thereby reducing the mobility of the polymer chains. Therefore, an increase in the amount of glutaraldehyde will result in a decrease in the elongation break value [8].



Fig. 3(C) shows that the increasing glutaraldehyde concentration decreases the degradation of bioplastics (Fig. 3(C)). The cross-linking between the active groups in the polymer and the aldehyde groups in glutaraldehyde enhances the structural strength of bioplastics, rendering them less susceptible to water absorption, thus inhibiting the biodegradation process of bioplastic [30].

### 3.4 Bioplastic Product

The results indicated that the optimum conditions to produce bioplastics were the glycerol addition of 1%, the cellulose/chitosan ratio of 25:75, and the glutaraldehyde addition of 1%. The product appearance and FT-IR Spectrum of bioplastic that was produced from optimum conditions are shown in Fig. 4



**Figure 4. (A) Physical appearance and (B) FT-IR Spectrum of bioplastic product (optimum condition)**

The chemical structure of bioplastic products from raw chitosan and corn-stalk fiber under the optimum conditions was analyzed by FTIR spectroscopy. Fig. 4 showed that at wavenumber  $3323\text{ cm}^{-1}$ , indicating the presence of an OH group from cellulose and chitosan [21]. Absorption of CH groups ( $\text{CH}_3$  asymmetric stretching) was observed at a wavenumber of  $2901\text{ cm}^{-1}$ , amine groups from chitosan at  $1582\text{ cm}^{-1}$  [20], and cellulose CH groups ( $-\text{CH}_2-$  bending asymmetric) at  $1400\text{--}1500\text{ cm}^{-1}$  [6]. The wavenumber of  $1368\text{ cm}^{-1}$  indicates the C=N group due to the interaction of chitosan with glutaraldehyde. The CO glycosidic bond is shown in wavenumber  $1024\text{ cm}^{-1}$  [31]. The wavenumber of  $895\text{ cm}^{-1}$  indicates the presence of CO strain from the cellulose component [12].

## CONCLUSION

The introduction of glycerol can increase the swelling degree, elongation at break, and the rate of bioplastic degradation. However, the addition of glycerol can reduce the tensile strength of bioplastic. Increasing the cellulose concentration in cellulose/chitosan bioplastics can cause a decrease in the swelling degree, an increase in tensile strength, an increase in elongation break, and enhanced biodegradability. The crosslinking agent of glutaraldehyde causes a reduction in the degree of swelling, an increase in the value of tensile strength, a decrease in the value of elongation break, and a decrease in the biodegradability of bioplastic. The optimum conditions to produce the best bioplastics were the glycerol addition of 1%, the cellulose: chitosan ratio of 25:75, and the glutaraldehyde addition of 1%.

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