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The Effect of Chitosan Concentration on the Characteristics of Sago (Metroxylon sp) Starch Bioplastics.

Tuty Anggraini*, Ulfimarjan, Fauzan Azima, and Rina Yenrina.

Agricultural Product of Technology, Faculty of Agricultural Technology, Andalas University 25163.

ABSTRACT

This study aimed to determine the effect of chitosan concentration on the characteristics of sago starch bioplastics. This study used a completely randomized design consisting of 5 treatments and 3 repetitions. Data were analyzed statistically using ANOVA and continued by Duncan's New Multiple Range Test (DNMRT) at the 5% significant level. The treatments in this study are the differences in concentration of chitosan added at 0% (as a control), 5%, 10%, 15% and 20%. The results showed that the increased concentrations of chitosan on bio plastics affect the thickness, density, water absorption, tensile strength, elongation, thermal and morphological properties of bioplastics. Increasing concentrations of chitosan tends to increase the value of thickness, density, tensile strength, but it decreased the value of water absorption and elongation of bioplastics. Biodegradation test showed the difference of chitosan concentration of bioplastics with no significant effect. The best bioplastics obtained in this study is a bioplastic added with chitosan concentration of 20% with a thickness of 0.106 mm; density of 1.41 g / cm3; water absorption 130.31%; tensile strength of 46.71 MPa; and elongation of 0.32%.

Keywords: bioplastics, chitosan, sago starch.

*Corresponding author



INTRODUCTION

The use of plastics as a food packaging material has increased considerably. Plastic is one of the materials used for the needs of household tools, packaging materials, and so forth [1]. However, the use of plastic is bad for the environment, the waste generated each day is biodegradable and it requires a long time to degrade. Bioplastic is a form of plastic that can be derived from biological resources and are biodegradable.

Bioplastics can be used just like conventional plastic, but it will be destroyed discomposedly by microorganism's activity to the outcome of the water and carbon dioxide after it is used up and discarded into environment. Another advantage of bio-plastic materials is; it can be renewable and the amount is abundant. While, starch is one of the materials most widely used and promising in the bioplastics market for biodegradability, availability, more friendly and cheap [2].

Sago starch is one of potential sources to make bio-plastic. The potential production as well as the wide land of sago in Indonesia is very large but only few are being utilized. According to data from the Kementrian Pertanian [3], it estimated that the sago land area of 2.5 million hectares located in Indonesia with 1:25 million hectares (50%), and of the 1.2 million ha area located in Papua and West Papua. Sago starch consumption in Indonesia is only about 210,000 tons or 4-5 percent of potential new production [4].

According to Averous [5], starch-based bioplastic has the disadvantage of low physical properties, brittle, low resistance to water due to the hydrophilic of the starch. One way to improve the properties of bioplastics starch is by the addition of chitosan. Chitosan is a polymer that can be added to the starch-based bioplastics, it is due to the hydrophobic of chitosan compared to starch. Besides, chitosan is also nontoxic, biocompatible and biodegradable, making it safe to be used. Chitin is found from natural sources such as the shell and the head of group of *crustacea* / crabs and shrimp [6].

Chitosan can be extracted from chitin through conventional extraction and enzymatic reaction. Conventional methods of chitosan's extraction from shrimp shells include four stages in which *deproteination*, removes proteins through alkali treatment, demineralization removes calcium carbonate and calcium phosphate by acid, depigmentation removes pigment through bleaching procedures and de-acetylation to change chitin into chitosan by using an alkali solvent [7]. Shean *et al* conducted research on the manufacture of bioplastics from potato starch with the addition of chitosan. Chitosan was added to the potato starch at variety of 5, 10, and 15% by the weight of starch. The result of chitosan concentration of 15% has a good mechanical strength [8]. In this study, the authors firstly conducted a preliminary research; it showed that the addition of chitosan concentration of 20% was still able to form a good plastic film. Therefore, in this study, chitosan concentration used was 5%, 10%, 15%, and 20% by the weight of starch.

MATERIAL AND METHODS

The materials used in this study were obtained from the sago starch at the area of Koto Marapak, Pariaman, West Sumatra. Chitosan with the de-acetylation degree of 75% will be synthesized from the shrimp shell, HCl, NaOH, glacial acetic acid 98%, Sorbitol, 95% ethanol, a solution iodine and distilled water.

The equipment used: thermometer, hot plate, analytical balance, oven, glass mold 20x20x0,2 cm, vernier caliper, while for the purposes of quantitative and qualitative analysis, it used Universal Testing Machine (UTM) - COM TEN Series 95F, UV Spectrophotometer 1800 and Fourier Transform Infra Red.

A.Chitosan Preparation

Chitosan that used in this study was made from shrimp shells obtained from the traditional market in Purus, Padang. The method of making the chitosan refers to research conducted by Suptijah [9] where shrimp are separated from their shells and meat. Shrimp shell is removed, cleaned and dried in the dryer cabinet for 24 hours, the dried shrimp shells that have been crushed to a size of 20 mesh. Shrimp shells are demineralized by using 1.5 N HCl with solids and solvent ratio of 1: 7. Temperature used in the process of demineralization was 90 ° C for 2 hours. After the demineralization process complete, shells are washed by using distilled water until it reaches a neutral pH. Furthermore, a process of deproteination used 3.5 N NaOH with solids and solvent



ratio 1:10. Temperature used in the process deproteination is 90 minutes for 2 hours. After that, the shells are washed up to a neutral pH. The latter process is the process of de-acetylation wherein the shrimp shells removed its acetyl group using 50% NaOH, with a temperature of 140 ° C for 1 hour with the solids and solvent ratio of 1:10. Once the process is completed, shells washed until pH neutral and dried to obtain chitosan powder.

B. Bioplastics Preparation

Starch, sorbitol and chitosan were prepared according to treatment. Acetic acid solution 1% was prepared in 100 ml of solution. Chitosan was put in a 1% acetic acid while stirring it with a magnetic stirrer and heated on a hotplate with temperature 50° C to be dissolved perfectly in acetic acid. Starch and sorbitol which have been weighed incorporated into the chitosan solution was then stirred until homogeneous and thickens at a temperature of 70° C, then it was poured into glass plates with a size 20x20x0,2cm and dried it in an oven at a temperature of 40° C for ± 12 hours. After drying, bio-plastics is released from the glass plate.

The Formulation of Making Bio-plastics can be seen in Table 1.

| No. | Matarial | Treatments | | | | |
|-----|---|------------|----------|----------|----------|----------|
| NO. | Material | Α | В | С | D | E |
| 1 | Solution of chitosan in acetic acid | 0% | 5% | 10 % | 15 % | 20 % |
| 2 | Sago Starch (g) | 5 | 5 | 5 | 5 | 5 |
| 3 | Sorbitol (g) | 1,2 5 | 1,2 5 | 1,2 5 | 1,2 5 | 1,2 5 |
| 4 | Distilled water (ml) | 100 | 100 | 100 | 100 | 100 |

Table 1. Formulation Ingredients in Making Bio-plastics

C. Sago Starch Characterization:

1). Water Content [11]

Aluminum cup dried in an oven at 105°C for 15 minutes, then it is cooled and weighed (A). The sample is weighed as many as 5 grams (B). After that, the cup containing the sample was dried at 105°C for 6 hours and then it is cooled in a desiccator and weighed to obtain a fixed weight (C). The water content was calculated using the formula:

Water Content (%wb) =
$$\frac{[B - (C - A)]}{B}x 100\%$$

2). Preparation of Amylose Standard Curve : [11]

40 mg of pure amylose is weighed and put into a test tube, added 1 ml of 95% ethanol and 9 ml of NaOH 1 N. The reaction tube was heated in boiling water for approximately 10 minutes, until all the amyloses form a gel. Then, moved the entire mixture quantitatively into a 100 ml flask. Pipette each 1,2,3,4, and 5 ml of solution above and insert into each 100 ml flask. Added to each flask 1 N acetic acid by 0.2, 0.4, 0.6, 0.8, and 1.0 ml, then added each 2 ml of iodine solution. Adjusted mixture with distilled water, then, incubated for 20 minutes. The intensity of blue color formed was measured with a spectrophotometer at a wavelength of 625 nm.

Amylose content [11]

A sample of 100 mg was weighed, put it into a test tube, added 1 ml of 95% ethanol and 1N NaOH. The sample was heated for 10 minutes to gelatinize starch. Once it is cooled, put the pasta starch in 100 ml

Source: Modified from Erfan [10] Description: The percentage of taken chitosan concentration of the weight of sago starch



and adjust with distilled water. As many as 5 ml are pipetted and put it in a 100 ml and then added 1 ml of acetic acid 1 N, 2 ml solution of iodine and distilled water, shake incubated for 20 minutes. An absorbance of the sample was measured with a spectrophotometer at a wavelength of 625 nm and amylose content calculated using the formula:

Amylose (%) = $\frac{C \times V \times FP \times 100\%}{\text{weight of the sample (mg)}}$

C = Concentration of amylose samples from the standard curve (mg / ml) V = Volume of final sample (ml) FP = Dilution Factor

D.Chitosan Characterization

Degree of deacetylation [6]

Determining the degree of de-acetylation of the chitosan using FTIR spectroscopy is done in the following way: chitosan made into pellets with KBR to form a thin layer of transparent. Furthermore, the uptake was measured by FTIR with a frequency of 400 to 4000 cm-1. Degree of de-acetylation is calculated by the equation:

% Degree of de - acetylation =
$$1 - \left[\frac{A_{1655}}{A_{3450}}x\frac{1}{1,33}\right]x$$
 100%

E. Bioplastics Characterization

1). Water Absorption [12]

The sample used in the form of sheets measuring 3 inches x 1 inch x thickness of materials. The samples are previously dried for 24 hours in an oven at a temperature of 50° C, cooled it in a desiccator and immediately weighed. The data of water absorption was obtained by soaking the samples in water for 2 hours. Then the sample was dried with a cloth and immediately weighed.

- water absorption (%) = [weight gain (%)+solute loss (%)]

 Addition of weight (%) = weight after immersion (g) – Initial weight (g)) Initial weight
 x100%

 Material Dissolved missing (%) = <u>Initial weight (g) – Weight after drying and soaking (g))</u> (Initial weight (g))

2). Thickness Measurement [13]

Thickness of bioplastic was measured using a micrometer thickness gauge at five different places. The thickness value was measured from the average of five measurements of bioplastic.

3). Tensile Strength [13]

A bioplastic that will be analyzed is cut with a length of 50 mm and a width of 10 mm. After that, the specimen is clamped on universal tensile testing apparatus and pulled at a constant speed and a maximum load of 5 kg. From the values obtained, it can be determined the amount of tensile strength using the equation:

σ = Fmaks / A
 σ = tensile strength (MPa)
 Fmaks = maximum Force (N)
 A = cross-sectional area (mm2)



4). Elongation [13]

Elongation measured by the equation:

 $\% E = \Delta L / Lo$ % E = Elongation (%) $\Delta L = accretion of specimen length (mm)$ Lo = initial specimen length (mm)

5). Density of Bioplastics [14]

Tests conducted on the density of the sample size of 5 x 5 cm, Sample is tested weighed and measured dimensions that the thickness at five different points. Calculation of density:

Density $(g/cm^3) = \frac{\text{Sample Weight }(g)}{\text{Volume of Sampel }(cm^3)}$

6). Thermal Properties

Testing the melting point was done by using an oven with a temperature variation 100° C - 250° C. Samples measuring 1x1 cm put in the oven for 10 minutes.

7). Morphological Structure

Bioplastic film morphology test performed using a microscope Trinocular. This test aims to determine morphological differences films with different concentrations of chitosan. Testing was done by cutting the film with a size of 1×1 cm, and then put the samples over the preparation after it was observed by using a microscope so that the structure of the film surface bio-plastics can be seen.

8). Biodegradation Test [15]

Biodegradation test was performed by the method of burial in the ground. The simplest quantitative method to characterize the occurrence of a polymer biodegradation is to determine the mass loss and polymer material degradability. The loss of mass is determined by weighing the mass of the polymer before and after biodegradation process during a certain time of the interval. The testing biodegradation of plastic film is done in vitro in environmental media such as soil in accordance with standard methods recommended by the American Society for Testing and Materials (ASTM). In this experiment, the land will be taken from the Flower Garden area in Khatib Sulaiman, Padang. Parameter decomposition observed is a reduction in the weight of the plastic being tested after the burial (Burial Soil Test) in the ground.

Each sample is inserted into a plastic tray. Plastic film with a size of 2 cm x 3 cm from each treatment was put in the ground (\pm 3cm). After a certain period of samples taken from the test sites, it is cleaned with distilled water and dried to constant weight, and then weighed the remained heavy plastic film. The period of time to making plastic film were the first week, second, third, fourth, and fifth week.

Mass loss (%) = $\frac{W_2 - W_2}{W_1}$ W₁ = mass of bioplastic before degradation W₂ = mass of bioplastic after degradation

9).Statistical Analysis

A completely randomized experimental design was used to study the chitosan effect on the properties of bioplastics. Analysis of variance (ANOVA) was used to determine the effect of chitosan. If difference in mean existed, multiple comparisons were performed used Duncan's Multiple Range Test (DNMRT).

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RESULTS AND DISCUSSION

A.Characteristics Sago Starch

In this study, sago starch obtained from Marapak Koto area, Eastern District of Pariaman, Pariaman. Starch obtained in wet conditions with water content of 45%. Sago starch is dried and characterized the water content and the ratio of amylose. Sago starch characterization results after drying can be seen in Table 2

Table 2. Characteristics of Sago Starch

| Parameter | Amount |
|-------------------|--------|
| Water Content (%) | 10,34 |
| Amylose (%) | 23 |

In this study, the water content obtained after drying was 10.34%. The water content of sago starch used in this study is lower than the water content of sago starch used [16] in the manufacture of bioplastics that is equal to 11.58%. The water content is an important component in a material that can affect the quality of the material itself or the resulting product. The water content of sago starch is determined by both processing process and drying process. The water content of sago starch is very important because it deals with the storage time of the sago starch itself.

Table 2 showed that the ratio of amylose used as many as 23%. The low levels of amylose are influenced by different sources sago starch is used and its growth place. According to Tri [17], the higher amylose content will be obtained by bonding the film structure where hydrogen bond between chains becomes more robust. Amylopectin branched polymer structure - a branch causes the distance between the chains is relatively further away so that the hydrogen bonds between chains that are formed becomes weaker. Therefore, the strength of starch films is more determined by the content of amylose.

B. Characteristics of Chitosan

In this study, chitosan is synthesized from shrimp shell waste through the demineralization process, deproteination, and de-acetylation. Chitosan is obtained characterized and uses as a treatment of making bio-plastics. Chitosan characterization results generated can be seen in Table 3.

| Parameter | Amount | Sugita <i>et al</i> (2009)* |
|--------------------------|--------|--------------------------------|
| Water Content (%) | 6,3 | ≤ 10 |
| Ash Content (%) | 0,36 | ≤ 2 |
| Degree of de-acetylation | 75 | ≥ 70 |
| (%) | | |
| The Yield (%) | 22,4 | - |

Table 3. Characteristics of Chitosan Produced Parameter Number [6] *

The water content is a very important parameter for determining the quality of chitosan. The water content influenced by the process of drying, drying time, and the surface area of a dried chitosan. The water content of chitosan based on Sugita [6] was below 10%, Table 3 showed that the results of the water content of chitosan produced. The chitosan produced had water content of 6.3% thus the water content of chitosan meets standards quality of chitosan.

The ash content is a parameter to determine the minerals contained in the ingredients that characterizes success demineralization process is done. According to Sugita *et al* [6] chitosan ash content must be below 2%. Based on the analysis, ash content chitosan obtained comply with quality standards chitosan that is equal to 0.36% of the requirements of a maximum of 2%. This suggests that the chitosan obtained has a good quality.

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Description: mark * indicates the quality standards of chitosan by Sugita et al [6]



One quality parameter of chitosan is the degree of acetyl. In this study, the use of chitosan degree of de-acetylation 75% .The higher degree of acetyl showed the higher purity of chitosan, which means that chitosan is already pure of impurities, which are proteins, minerals, and the acetyl group [9]. Degree of de-acetylation shows the percentage of the acetyl group which can be removed from the chitin to produce chitosan. The high degree of de-acetylation indicates that the acetyl groups contained in the low chitosan. The quality standards for the degree of de-acetylation of chitosan by [6] was greater than 70%, when compared to chitosan in the chitosan study that meets quality of chitosan standards.

C. Characteristics of Bioplastics

1). The thickness of Bioplastics

The thickness is one of the important parameters that influence the bioplastics. The value of the average thickness of bioplastics for each treatment was presented in Table 4.

| Treatment | Thickness (mm) (Mean ± Standard Deviation) | | |
|----------------------|---|--|--|
| A (Without chitosan) | 0,089 ±0, 0005a | | |
| B (Chitosan 5%) | 0,091 ±0,0041 a | | |
| C (Chitosan 10%) | 0,099 ±0,0023 b | | |
| D (Chitosan 15%) | 0,103 ±0,0017 bc | | |
| E (Chitosan 20%) | 0,106 ± 0,0071 c | | |
| Coef of Var = 3,35 % | | | |

Table 4. Average values Thickness Bio-plastics from different treatment

Description: The figures in the column, followed by lowercase letters are not the same, significantly different according DNMRT test at 5% level.

Based on the analysis of variance, showed that the different chitosan concentration influences significantly at 5% level. Table 4 showed the differences in the thickness of the bioplastics value with the addition of chitosan composition variation, wherein the thickness of bioplastics will further increase with the increasing in the addition of chitosan concentration. Table 4 showed the thickness of bioplastic-ranged from 0.089 mm to 0.106 mm. The highest thickness of bioplastic is in the treatment E (chitosan 20%) with an average thickness of 0.106 mm and the lowest thickness was in the treatment of A (without chitosan) with an average thickness of bioplastics will increase. The thickness of bioplastics were influenced by the amount of total solids in solution as well as spacious and volume of the solution in the mold. The more total solids contained in the solution then the bio-plastics produced become thicker [18].

2). Density of Bioplastics

According to Tipler [19] the density is defined as the ratio of the weight of each sample to test against volume. The higher the density value the better of the density of the bioplastics becomes. The density of each treatment can be seen in Table 5.

| Treatment | Density (g / cm3) (Mean ± Standard Deviation) | |
|----------------------|--|--|
| A (Without Chitosan) | 1,32 ± 0,008 a | |
| B (Chitosan 5%) | 1,33 ± 0,003 a | |
| C (Chitosan 10%) | 1,34 ± 0,004 a | |
| D (Chitosan 15%) | 1,36±0,022 b | |
| E (Chitosan 20%) | 1,41±0,033 c | |
| Coef of Var = 0,85% | | |
| | | |

Table 5. Average Density Bio-plastic value of different treatments



Description: The figures in the column, followed by lowercase letters are not the same, significantly different according DNMRT test at 5% level.

The addition of chitosan increased the density of bioplastics. This is caused by the increased regularity of the arrangement of molecules in the alloy. However, the increased concentrations of the chitosan did not significantly increase the density of bio-plastics in the treatment A, B and C. The density is also influenced by the value of the degree of crystallinity. Amorphous molecular structure has a relatively low density of the crystalline molecules. The increasing of the density of molecules is caused by the degree of crystalline bioplastics increased concurrently with the addition of chitosan [20].

According to Wan *et al* [21] the structure of the chitosan crystallinity increases with the degree of deacetylation of chitosan. In the present study, the degree of de-acetylation of chitosan used was 75%. The high degree of de-acetylation resulting in the crystalline structure of chitosan increased. The addition of a polymer having a high crystalline properties in bio-plastics led to an increase in the density of bio-plastics. The structure of crystalline polymers which has higher densities because of the structure of the crystalline polymer has a more regular arrangement so that it has a higher density. In general, a polymer that has high crystalline has a high density as well [22].

3). Water Absorption of Bio-plastic

Water absorption test was conducted to determine the effect of chitosan on the water absorption of sago starch bioplastics. Water absorption value of each treatment can be seen in Table 6.

| Treatment | Water absorption (%) (Mean ± Standard Deviation)) | |
|----------------------|---|--|
| A (Without Chitosan) | 204,97 ± 3,92 e | |
| B (Chitosan 5%) | 193,27 ± 1,29 d | |
| C (Chitosan 10%) | 175,91 ± 5,23 c | |
| D (Chitosan 15%) | 154,28±5,72 b | |
| E (Chitosan 20%) | 130,31 ± 2,88 a | |
| Coef of Var = 2,38% | | |

Table 6. Average Values Water Absorption Bio-plastic From Different Treatment

Description: The figures in the column, followed by lowercase letters are not the same, significantly different according DNMRT test at 5% level.

The addition of chitosan significantly affects to water absorption of bioplastics. Table 6 showed the difference in absorption of water by the bioplastics value with the addition of chitosan composition variation, wherein the water absorption of bioplastics will decrease with the increased addition of chitosan concentration. It can be seen that the absorption of water by the bioplastics ranged from 204.97% to 130.31%. The absorption of water by the lowest bioplastics is in treatment E (chitosan 20%) with the absorption value of 130.31% and the highest water absorption is in the treatment of A (without chitosan) with the percentage of water absorption by 204.97%. From these results, it can be concluded that the higher the concentration of chitosan, the decreased the ability of water absorption of bioplastics. Water absorption in bioplastics is affected by density. Increasing the density of molecules caused by the degree of crystalline bioplastic is increased so that the absorption of water by the bioplastics is decreasing [20].

4). Tensile Strength

Tensile strength is important mechanical characteristics tested to determine the quality of bioplastics. Tests carried out using ASTM standard with a thickness of 0.1 mm \pm bio-plastics. The measurement results of tensile strength against the bio-plastic can be seen in Table 7.



| Treatment | (Pull Strong (Mpa) (Mean ± Standard Deviation) | | |
|----------------------|---|--|--|
| A (Without Chitosan) | 23,86 ± 2,52 a | | |
| B (Chitosan 5%) | 30,20 ± 1,10 b | | |
| C (Chitosan 10%) | 38,83 ± 3,53 c | | |
| D (Chitosan 15%) | 42,58 ± 2,22 c d | | |
| E (Chitosan 20%) | 46,71 ± 2,99 d | | |
| Coef of Var = 7,28% | | | |

Table 7. Average values Strong Pull Bio-plastic From Different Treatment

Description: The figures in the column, followed by lowercase letters are not the same, significantly different according DNMRT test at 5% level.

The addition of chitosan concentration on bioplastics has different effects on the strenght of bioplastics produced. The values for tensile strength bioplastic in this study ranged from MPa 23.86 - 46.71 MPa. The highest tensile strength is in treatment E (Chitosan 20%) of 46.71 MPa and the lowest tensile strength is for the treatment of A (without chitosan) in the amount of 23.86 MPa. It can be concluded that the increasing concentrations of chitosan causes the increased value of the tensile strength of bioplastics produced. Tensile strength grades of bioplastics produced higher when compared with the research [8] tensile strength bioplastic potato starch / chitosan produced ranging from 30 MPa - 40 MPa. The Values for the strength tensile of bio-plastic in this study was similar to the strength tensile synthetic plastics such as polypropylene where its strength reached 41 MPa [23]. Therefore, it can be concluded that the tensile strength bioplastic value of this research has great tensile strength values.

The addition of chitosan concentration causes the changes in the mechanical properties of bioplastics. It occurs because the mechanical properties are affected by the large amount of content of the constituent components bioplastics (starch, sorbitol and chitosan). Chitosan as additional polymers will provide the rigid nature film that causes tensile strength in bio-plastics is increasing. The addition of chitosan will increase the amount of solid component in bio-plastic formulations in which increasing amount of solids in the bioplastics will lead to the ability sorbitol as plasticizer decline. It leads to a decreased ability of sorbitol causes the attractive force of intermolecular become stronger so that the value will be higher in the tensile strength [18].

5). Elongation

Elongation is the percentage of bioplastics extension after the plastic was cut by the influence of the applied force. Based on analysis of the value of the elongation of bioplastics can be seen in table.8

| Treatment | Elongation (%) | | |
|-----------------------|-----------------------------|--|--|
| Treatment | (Mean ± Standard Deviation) | | |
| A (Without Chitosan) | 1,76 ±0,20 d | | |
| B (Chitosan 5%) | 1,32 ±0,10 c | | |
| C (Chitosan 10%) | 0,92 ±0,04 b | | |
| D (Chitosan 15%) | 0,78 ±0,02 b | | |
| E (Chitosan 20%) | 0,32 ±0,08 a | | |
| Coef of Var = 10,97 % | | | |

Table 8. Average Elongation Bioplastics From Different Treatment

Description: The figures in the column, followed by lowercase letters are not the same, significantly different according DNMRT test at 5% level.

Based on the analysis of variance, it showed that the differences in the concentration of chitosan significantly affected based on the table of variance of 5% to the elongation of sago starch bioplastics. Table 8 showed the difference in elongation bio-plastics value with the addition of chitosan composition variation, where the elongation of bio-plastics will decrease with the increased addition of chitosan concentration. From the analysis results, it obtained the percent extension of sago starch bio-plastic with chitosan variation

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between 0.32% - 1.76%. The lowest bio-plastics elongation value in treatment E (chitosan 20%) has a value of 0.32% elongation and high elongation on treatment A (without chitosan) with a value of elongation of 1.76%.

The addition of chitosan significantly effect on the percent extension of bioplastics, but its impact tends to be negative. Bioplastics elongation value as shown in Table showed the increasing concentrations of chitosan. The same result is shown from Lopez *et al* [22] in which the elongation value obtained by the 2.36% to 1.64%. The addition of chitosan at 10% resulted in a decrease in the elongation of bio-plastics. It is connected with the nature of the crystallinity of chitosan is added where the properties of chitosan crystalline can caused the structure bio-plastic being organized and thus lowering the elongation of bioplastics.

6). Thermal properties

The tests are conducted to determine the thermal properties of the warming effect of sago starch bio-plastics variety of different chitosan. Observations of temperature of different bio-plastics can be seen in Figure 1.

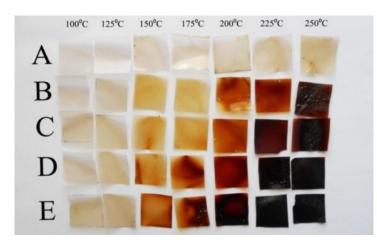


Figure 1. Bioplastics after heating

In Figure 1, it can be seen that the higher the concentration of chitosan, then bioplastics produced will be increasingly vulnerable to warming temperatures. This is according to research conducted by Lopez *et al* [22] in which the thermal properties of bioplastics, the results show more and more every additional chitosan on starch polymer matrix will result in bioplastics will be increasingly vulnerable to warming.

The increase in heating temperature not only affects the appearance of bioplastics but also will affect the mechanical properties of bioplastics, which with increasing warming temperatures will result in bioplastics increasingly fragile and easily broken. Heating temperature of 100 ° C for 10 minutes on bioplastics can be a bit fragile. At a temperature of 125 ° C, bioplastics start to be brittle and easily broken. A decrease in mechanical properties is increasing so that the color produced bioplastics may become darker.

The decline of the bioplastics resistance heating temperature is caused by the interaction of chitosan and starch of bioplastics. When chitosan is added on bioplastic formulations, the polymer chitosan and starch will form hydrogen bonds between the hydroxyl groups of starch and amino groups of chitosan. But the result of interaction between starch chains weakened and the starch molecules are not in the optimal position, interactions among starch are weak and little energy is required to the bioplastics damage [22]. From these data, it can be concluded that the high heating temperature will have a negative effect on the bioplastic so that it can decrease the mechanical strength. Therefore, the application of bioplastics sago starch is limited to a certain temperature.

7). Structure Morphology



The tests conducted using a microscope to see the spread of chitosan on sago starch bioplastics such as the structures can be a general overview of the quality of mixing, dispersion of materials in sago starch bioplastics and the forecast processes that occurs in bio-plastics. Testing of bioplastics's surface morphology was performed with a microscope at a magnification of 10 times, the surface morphology test results are presented in Figure 2.

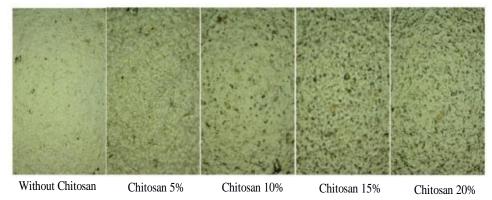


Figure 2. The Distribution of Chitosan Viewed by Microscope Trinocular At 10x magnification

In Figure 2, it is seen the change in morphology of the sample with the addition of chitosan concentration. Chitosan is added to bioplastics spread to all parts of bioplastic as a growing number of chitosan are added, the more loopholes can be filled by chitosan. Then it causes bioplastic has a higher density and tensile strength to be increased. Based on Lopez *et al* [22], mixing starch and chitosan produce a smooth surface structure without pores, homogeneous and flat. Bonilla conducted research on the film formation wheat starch with the addition of chitosan, results show that the structure of the film that forms a uniform, mixing starch and chitosan form a homogeneous mixture and no pores or cracks were observed [24]. Therefore, it can be concluded that the addition of chitosan on starch bioplastic formulation will make the surface structure of bioplastic is getting better.

8). Biodegradation Tests

One of the most important properties of bioplastics is its ability to be degraded naturally in the environment so that the damage caused by the use of synthetic plastic does not occur. Biodegradation is one parameter observation that might indicate that bioplastics are environmentally friendly or not. The simplest quantitative method to characterize the occurrence of a polymer biodegradation is to determine the loss of mass during testing.

Tests carried out on each treatment with three replications. The biodegradation testing conducted on a sample size of 2x3 cm. Each sample is inserted into a plastic tray that has a length of 34 cm, width 28 cm and depth of 13 cm. Land that is used as a medium of biodegradation taken in the area Khatib Sulaiman, Padang. Before being used as a medium of biodegradable bio-plastics, soil is measured its water content and pH. The water content of the soil used was 60.47% and had a pH of 6.2. Sample mass reduction is seen every week for one month at room temperature. Based on analysis of biodegradable bio-plastics value, it can be seen in Table 9.

| Treatment | Mass Loss (%) | | | |
|-----------|---------------|--------|--------|--------|
| | Week 1 | Week 2 | Week 3 | Week 4 |
| А | 22,03 | 31,52 | 44,59 | 47,96 |
| В | 17,42 | 40,93 | 51,61 | 54,06 |
| С | 23,41 | 43,45 | 50,36 | 50,44 |
| D | 15,71 | 36,52 | 47,29 | 53,67 |
| E | 21,45 | 40,07 | 47,11 | 51,43 |

Table 9. The average value of the mass loss on biodegradation testing bioplastics for 4 weeks



In Table 9, it shows that the addition of chitosan to the characteristics of sago starch bioplastics was not significantly different at 5% level. The addition of chitosan did not affect the rate of biodegradation of bioplastics due to chitosan is also a biodegradable polymer [6].

In this test, it shows that the starch polymer and chitosan used in bioplastics proved to be biodegradable, as seen in the first week, losing mass ranges bio-plastics 15.71% - 22.03%. In the second week, the mass loss ranging bioplastics due to the burial is at 31.52% - 43.45%. The mass reduction of bioplastics continues to increase the length of time the biodegradation testing. It shows the bio-plastic could be degraded in soil media. But the biodegradation testing week 3 and 4 show that the presence of a significant mass does not occur in each treatment bio-plastics. This is presumably because the soil moisture content is used as a medium of biodegradation decreases; the absence of controlling soil moisture content is used as a medium of mass loss causes biodegradable bioplastics at weeks 3 and 4 are not so significant.

CONCLUSIONS

The use of chitosan with various concentrations was significantly effected on the thickness, density, water absorption, tensile strength and elongation of bio-plastics. The addition of chitosan resulted to bio-plastics heat resistance tends to decrease and morphologically the process of making bioplastics by adding chitosan undergo the structural changes. The difference in concentration of chitosan had no effect on the biodegradation of samples bioplastics but the producing of bioplastic tends to decrease at each week of the observation.

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