

Synthesis of Bioplastics on Rice Straw Cellulose Using Orange Peel Extract, Chitosan, and Sorbitol

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Abstract

With the use of several plasticizers (orange peel extract, chitosan, and sorbitol), rice straw cellulose has been used to synthesize and characterize bioplastics. This study aims to synthesize and characterize bioplastics from rice straw cellulose with various additions of plasticizers, namely orange-peel extract, chitosan, and sorbitol, each with a certain concentration so that quality improvement can occur. The methods used in this study include testing the tensile strength value, testing the percentage elongation, and testing the percentage of biodegradation. In addition, functional group characterization tests were also carried out using FTIR. The results obtained in this study were the best values for tensile strength and percentage elongation in treatment A, namely 10.2611 MPa and 13.88%. The percentage of biodegradation for 7 days reached 50.58%. Functional group absorptions were found in various plasticizers, namely C-H, O-H, C-O, C-N, and C=C. Based on existing data, bioplastics synthesized using rice straw cellulose in various plasticizers meet JIS (Japan Industrial Standards).

Keywords: bioplastics; orange peel extract; rice straw; chitosan; sorbitol

Introduction

One of the biggest issues in the world is plastic pollution. Based on the data, 8.3 billion tons of plastic have already been produced. In 2015, almost 6.3 billion metric tons of plastic waste were produced; approximately 9% of it was recycled, approximately 12% of it was burned, and the remaining 79% ended up in landfills or the environment. By 2050, 12,000 tons of plastic waste might be in landfills or the

environment if plastic production and management continue exactly as they do nowadays (Geyer, Jambeck dan Law, 2017). Since plastic is so stable and long-lasting, it is frequently used in daily life. These characteristics also decrease environmental problems (Webb *et al.*, 2013). The environment will suffer tremendously from the buildup of plastic, especially aquatic organisms. Many helpless creatures eventually died after becoming tangled in

plastic debris or trapped in plastic nets (Hammer, Kraak and Parsons, 2012). These organisms can eat plastic waste. Furthermore, this waste has the potential to damage coral reefs (Gregory, 2009). Therefore, using plastics made from natural materials or bioplastics is one strategy used to decrease this problem. However, for bioplastics to decompose quickly in the environment, they must be biodegradable. Not all bioplastics are biodegradable, it should be highlighted (Ashter, 2016). This study focuses on the manufacture of biodegradables.

Because rice straw contains 32-47% cellulose, 19-27% hemicellulose, and 5-23% (Garrote, Domínguez and Parajó, 2002) (Saha, 2003), it has the potential to be used as a raw material for bioplastics (Bilo *et al.*, 2018). Moreover, the quantity of plentiful waste can be decreased by using rice straw as a raw material for bioplastics. According to reports, 1.6 kg of rice straw is produced for each kg of the crop during harvest. Because rice (grain) is the third largest production crop, the total amount of rice straw produced globally is about 1 billion tons (Maiorella, 1985). If this waste is not used, it will be burned or dumped in a river. Consequently, it will lead to greenhouse gas emissions, contamination, and pollution (Sangon *et al.*, 2018).

Numerous kinds of research on bioplastics have been conducted. Although there have been significant improvements, the quality is still poor. Bioplastics made from cellulose and starch have a stiff texture (Ndana dan Azizati, 2018). As a result, a plasticizer is used to continue developing bioplastics. A plasticizer is a substance used in the polymer industry as an additive (Sejidov, Mansoori and Goodarzi, 2005). There has been extensive research on bioplastics using glycerol plasticizers (Agustin dan Padmawijaya, 2016) (Coniwanti, Laila and Alfira, 2014), sorbitol (Afif, Wijayati and Mursiti, 2018), and orange peel extract (Rezki, Ratnawulan and Darvina, 2016). According to Afif *et al.* (2018), orange peel extract can increase the elasticity of bioplastics because it contains 94% limonene compounds. Both sorbitol and

orange peel extract function as plasticizers in bioplastics. Sorbitol is added as a plasticizer to bioplastics to increase elasticity and decrease stiffness.

Therefore, this study aims to synthesize and characterize bioplastics from rice straws using a variety of plasticizers, namely orange peel, chitosan, and sorbitol at certain concentrations. As a result, bioplastics' quality can be increased. (Pratiwi, Rahayu and Barliana, 2016) made bioplastics from rice straw by adding a combination of chitosan and cellulose pulp as a plasticizer. The highest tensile strength obtained was 4.2 MPa. (Inayati, Pamungkas and Matovanni, 2019) made bioplastics from rice straw with the addition of glycerol. The tensile strength and elongation values obtained were 6 MPa and 65%, respectively. (Susilawati, 2020) synthesized bioplastics from rice straw with a combination of chitosan and castor oil as plasticizers. Tensile strength, elongation, and Young's modulus values were 56.511 MPa, 16.140%, and 458.794 MPa, respectively. To the best of our knowledge, no one has yet used our proposed method to produce bioplastics made from rice straw.

Research Methods

Tools and materials

The tools and materials used in this research were the *Fourier transform infrared (FTIR)* (Thermo Fisher Scientific, US), the *Universal Testing Machine (UTM)* model UCT-5T (Orientec Co., Ltd., China), the autoclave (Thermo Fisher Scientific, US), the Memmert oven, the Cimarec *hotplate stirrer*, and the Bante *magnetic stirrer*. Rice straw (obtained from Patobong Village, Mattiro Sompe District, Pinrang Regency, South Sulawesi), and orange peels. whereas, the materials used were acetic acid p.a (Merck, Germany), hydrochloric acid p.a (Merck, Germany), methanol p.a (Merck, Germany), technical sodium bicarbonate (Mitra Wacana Media, Indonesia), sodium hydroxide p.a (Merck, Germany), chitosan (Sigma Aldrich, USA), and sorbitol.

Work Procedures

Cellulose Extraction from Rice Straw

The first step is preparing the rice straw which is ready for use. Three steps are required to produce cellulose extract from rice straw. The initial stage is dehydration, which aims to remove the water content. Cleaning rice straws entails washing them under running water and drying it afterward. Furthermore, the sieving process is carried out with a mesh size of 40 to reduce rice straw particles and improve maceration results. The second stage is maceration with methanol because this substance has polar properties that can attract protein, fat, and secondary metabolites present in the sample so that only cellulose and hemicellulose can be extracted. This stage is carried out for seven days by changing the solvent every 2×24 hours. This aims to maximally withdraw the compounds contained in rice straw (Monariqsa *et al.*, 2013). Fine samples are macerated using CH₃OH for a week. Although it is not enough for a week if the solution is colorless. The CH₃OH can be replaced or refilled. After the maceration process, the samples were filtered using Whatman filter paper no. 42. The residue from the filtering process is washed with water and dried. The third stage is delignification, which is carried out to remove the lignin content. The presence of lignin in rice straw will increase its stiffness (Pratiwi, Rahayu dan Barliana, 2016). It is caused by the polysaccharides consisting of cellulose and hemicellulose, not in a free state but being tightly bound to lignin. Then the addition of 17.5% NaOH to the residue was completely done until it was submerged. The mixture was heated to 121°C for sixty minutes. After that, refiltration is carried out, and the residue is also washed with water. The residue was then mixed with 2% HCl with the aim of hydrolysis, which was carried out for three hours. The process of washing, then drying, is carried out again in the oven (105°C) within an hour (Monariqsa *et al.*, 2013).

Orange peel extraction

Wash the orange peels thoroughly. Then soak them in a 5% NaHCO₃ solution for

10–14 hours with a ratio of 1:1. After that, the samples were mashed using a blender to produce more oil emulsions (Rezki, Ratnawulan and Darvina, 2016).

Making bioplastics films with the addition of orange peel extract and chitosan

As much as 0.8 g of chitosan was dissolved in 0.6 M CH₃COOH. After that, the solution was added along with a mixture of 0.8 g cellulose and 2 mL of orange peel extract. The mixture that formed was stirred using a magnetic stirrer for fifteen minutes and heated at 80 °C for seven minutes. After that, the mixture was poured and printed on a glass plate (Modification from Sumartono *et al.*, 2015).

Making bioplastics films by adding orange peel extract, chitosan, and sorbitol

As much as 0.8 g of chitosan was dissolved in 0.6 M CH₃COOH. After that, the solution was added along with a mixture of 0.8 g cellulose, sorbitol, and 2 mL of orange peel extract. The mixture formed is treated with the same treatment as before (Modification from Sumartono *et al.*, 2015).

Making bioplastics films with the addition of chitosan and sorbitol

As much as 0.8 g of chitosan was dissolved in 0.6 M CH₃COOH. After that, the solution was mixed with a mixture of 0.8 g of cellulose and sorbitol. The mixture formed is treated with the same treatment as before (Sumartono *et al.*, 2015).

Characterization of bioplastics

Tensile strength test and percent elongation

The bioplastic thin layer was formed into a cylinder. The voltage on the test equipment is set to 40 V and calibrate the tools then. The layer is then tested, and record the emerging data.

Functional group test

The thin layer was ground to a size of ±2 μm. The sample is then formed into pellets with a thickness of 0.3 mm (transparent). Then the pellet is put into the holder cell. After that, the instrument can be operated.

Biodegradation Test

The synthesized samples were stored in the soil for seven days. Observations were made for seven days with an interval of days 0; 3; 5; and 7. Then biodegradation test measurements are carried out.

Research Results and Discussion

Bioplastics made from rice straw cellulose

Table 1. Observations of variations in the addition of bioplastics

Bioplastics Variation	Color	Texture
A	yellow-brown	rather smooth
B	Brown	smooth and slippery
C	brown	Smooth and slippery

Notes:

A: with 2 mL of orange peel extract and 0.8 mL of chitosan

B: with 2 mL of orange peel extract, 0.8 ml of chitosan, and 2 mL of sorbitol

C: with 0.8 g of chitosan and 2 mL of sorbitol

Bioplastics A has yellow-brown color and a rather smooth texture. While B and C are brown, and the texture is not only smooth but also slippery. This difference occurs due to the addition of sorbitol and chitosan to B and C. The cellulose isolation process indicates the presence of lignin. The presence of lignin in rice straw will increase its stiffness (Pratiwi, Rahayu and Barliana, 2016). It is caused by the polysaccharides consisting of cellulose and hemicellulose is not in a free state but tightly bound to lignin. The 17.5% NaOH compound can be used to separate the lignin because this lignin is insoluble in simple solvents but soluble in dilute alkalis (Habibah, Nasution and Muis, 2013). This is supported by the statement (Safaria, Idiawati and Zaharah, 2013) that the OH in NaOH will break the bonds in the basic structure of lignin while Na⁺ will bind to lignin to form sodium lignat, which dissolves easily in water due to its polarity. A

black solution indicates the presence of dissolved lignin. At this stage, the temperature is 121°C. This aims to obtain finer-sized cellulose (Widayantini, Wirajana and Suarya, 2014). In this study, hydrolysis was carried out by soaking the residue for 1 hour using 5% HCl (Monariqsa *et al.*, 2013).



Figure 1. Bioplastics made from Rice Straw Cellulose

The cellulose produced is a yellow-brown powder. The results are following the research conducted by (Pratiwi dan Barliana, 2016).

Bioplastics Characteristics of Rice Straw Cellulose

Tensile strength and percent elongation

Table 2 shows that the results obtained to meet the JIS (Japan International Standard). The percent elongation value of treatment A was 13.88%, where the orange peel extract in A acted as a plasticizer. The hydrogen bonds formed are bound to the bioplastics and will form covalent bonds, which strengthen the hydrogen bonds that have been formed (Rezki, Ratnawulan and Darvina, 2016).

Table 2. Tensile strength test and percent elongation

Sample	Tensile strength (Mpa)	percent elongation (%)
A	10.2611	13.88
B	2.1441	13.54
C	1.5436	8.56

Based on the JIS, the value is already in the good category because it is more than 10%. The tensile strength value of treatment B was 10.2611 N/mm². The greater the addition of chitosan to the bioplastic, the more hydrogen bonds are formed in the bioplastic. It causes the chemical bonds

which contained bioplastics will be difficult to break, and requires a large amount of energy (Coniwanti, Laila and Alfira, 2014). The orange peel extract in the treatment acts as a plasticizer. The tensile strength value can increase the elasticity because orange peel extract can reduce hydrogen bonds and increase the intermolecular distance of the polymer. Consequently, it can bind particles in the empty spaces found in bioplastics (Rezki, Ratnawulan and Darvina, 2016). Based on JIS, the best value of tensile strength is 1-10 MPa.

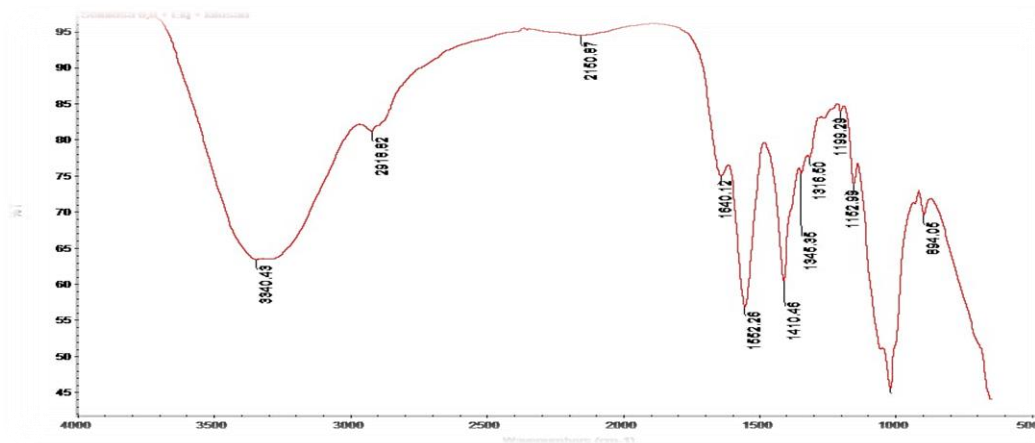
The percent elongation value of treatment B was 13.35%. The sorbitol addition can reduce the brittleness and increase the flexibility of bioplastics (Afif, Wijayati and Mursiti, 2018). Based on JIS, this value is in the fair category because it is

more than 10% (Epriyanti, Harsojuwono and Arnata, 2016). The tensile strength value for treatment B was 2.1441 N/mm². It still meets the JIS standard.

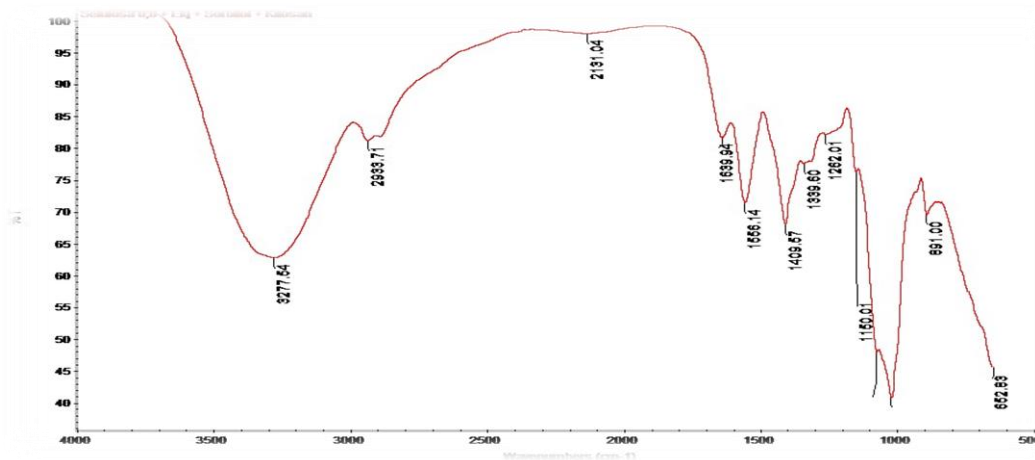
The percent elongation value for treatment C was 8.56%. Based on JIS, the value is close to the “good” category. The tensile strength value for treatment C was 1.5436. It also still meets the JIS standard. Treatment C showed differences from other treatments. It means the addition of 2 mL of sorbitol and 0.8 g of chitosan decreased the tensile strength and percent elongation. The tensile strength value of 1.5436 N/mm² is almost the same as the results obtained by Ndana dan Azizati (2018), which was 1.427 MPa.

Bioplastics Functional Groups

(a)



(b)



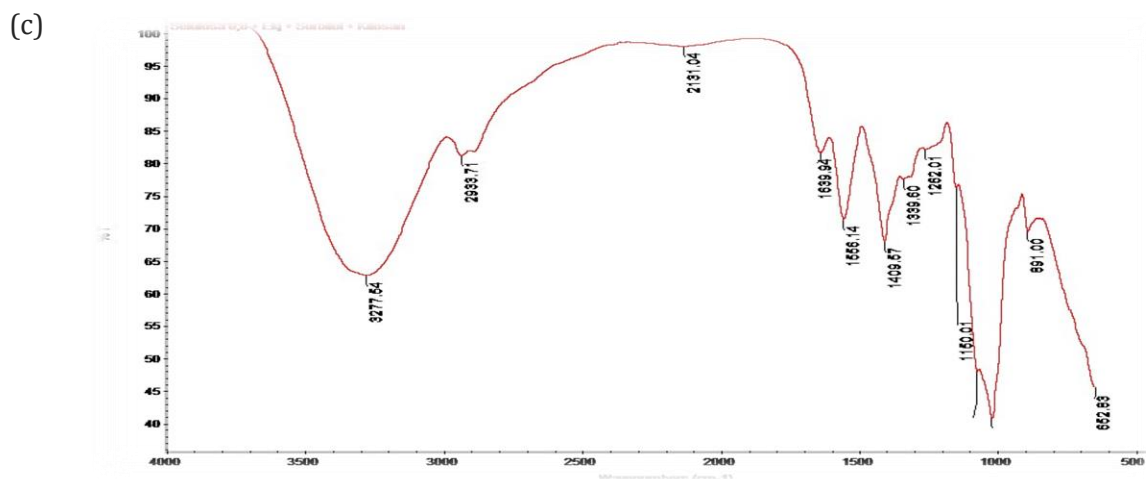


Figure 1. FTIR spectrum of rice straw cellulose bioplastics (a) orange peel extract and chitosan (b) orange peel extract, chitosan, and sorbitol (c) chitosan and sorbitol

Figure 1a shows specific groups. They are O-H, C-O, C-H, and N-H. The first group appears at wavenumber 3277.54, the C-O functional group appears at 1150.0, the C-H group appears at number 2933.71, and the N-H group appears at number 1556.14.

Figure 1b shows specific groups. The O-H group appears at number 3288.62. The N-H group appears at number 1562.37. The C-O group appears at 1149.42, and the C-H group appears at number 2932.00.

Figure 1c shows specific groups. The O-H appears at 3340.43. The C-O functional group appears at 1152.99. The C-H group appears at number 2918.81. The N-H group appears at number 1552.26. This is similar to the research results of Pratiwi *et al.* (2016). The C=C functional group which appears in the number 1640.12 is similar to the research by (Sarifudin, 2013).

The results of the characterization of these samples indicate the presence of cellulose. These results are as same as the research by (Pratiwi, Rahayu, and Barliana, 2016). Figure 1c (without the addition of orange peel extract) shows a lower

absorption compared to the addition of orange peel extract. In addition, the presence of aromatic C=C groups was seen in sample C without the addition of orange peel extract.

Percentage of biodegradation

In the bioplastics, biodegradation test exactly on sample A, it was observed that there was water absorption in the bioplastics on day 3. It caused an increase in the weight of bioplastic. On day 5, bioplastics started to degrade as much as 26.92%. On day 7, bioplastics have been decomposed by microorganisms in the soil which is merely similar to research by Rezki *et al.* (2016). The addition of orange peel extract makes bioplastics degrade more quickly by organisms in the soil. The obtained polymer with a low molecular weight can be decomposed by these microorganisms. In this process, polymer chains are broken which then produce organic compounds that are safe for the environment (Rezki, Ratnawulan dan Darvina, 2016).

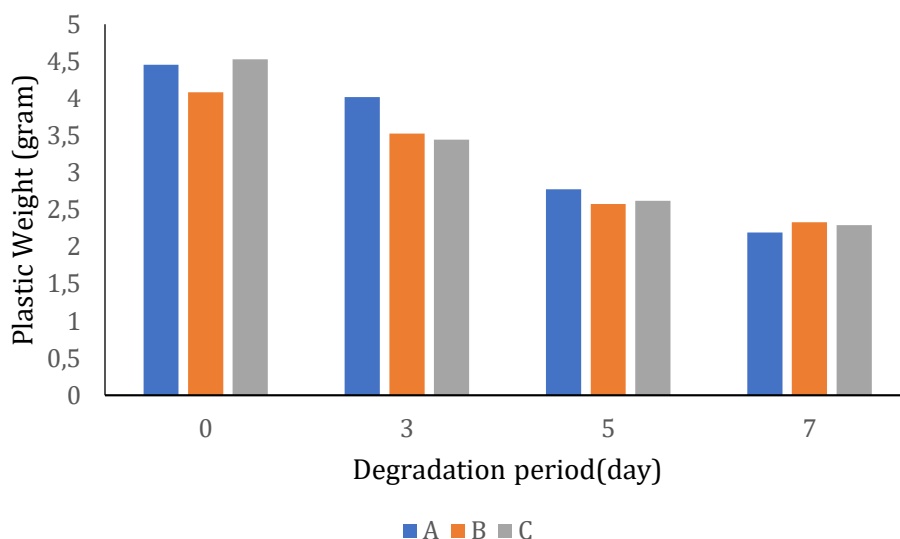


Figure 2. Graph of the relationship between degradation period and plastic weight

Figure 2 shows the results of the bioplastics biodegradation test. All samples have a decrease in weight. It describes that all samples can be degraded by soil bacteria. (Baharuddin, Rahayu and Febryanti, 2021) reported that isolate *S*₁₀ isolated from the soil adjacent to oil palm empty fruit bunch in East Luwu has the potential to degrade OPEFB waste which contains a high concentration of cellulose.

The percentage of cellulose biodegradation in treatment A on day 3 showed that there was water absorption in the bioplastics which caused weight gain. Treatment A starting from days 3 up to 7 had no signs of damage. The more chitosan was added, the slower the bioplastics were degraded by microorganisms (Hartatik, Nuriyah and Iswarin, 2014). According to the ASTM 5336 Standard for a 100% degradation rate, it takes 60 days for being degraded (Selpiana, 2015). It should be noted that the addition of chitosan makes it more difficult for bioplastics to decompose because chitosan is resistant to being decomposed by microorganisms in the soil. In addition, chitosan is difficult to dissolve in water in the soil because it has a natural hydrophobic reinforcement (Coniwanti, Laila and Alfira, 2014). Besides that, the density of intermolecular molecules in bioplastics causes the slowness of the process. It means that the decomposition of

enzymes produced by microorganisms is also slow (Selpiana, 2015).

Based on the data above, the biodegradation period of bioplastics with 2 mL orange peel extract and 0.8 g chitosan degraded the fastest after seven days of experimentation. On day 3, the percentage of biodegradation was only 9.74%. On day 5, the percentage increased by 37.70%. On day 7, the process of degradation by microorganisms in the soil reached 50.58%. The increase was due to the presence of functional groups hydrolyzed by enzymes. The more functional groups hydrolyzed by enzymes in soil microbes, the longer the biodegradation test. It can reduce the nutrients or compounds needed by microorganisms (Pratomo, 2011).

Conclusion and suggestion

Conclusion

Bioplastics can be made using rice straws with a combination of various plasticizers at certain concentrations. The best values of tensile strength and percent elongation were obtained in treatment A; 10.2611 MPa and 13.88%. The percentage of biodegradation for 7 days reached 50.58%. Some functional group absorptions found in various plasticizers are C-H, O-H, C-O, C-N, and C=C. Characterization data shows that the production of these bioplastics meets the Japanese Industrial Standard (JIS).

Suggestion

The next researchers may increase the mechanical tests and carry out bioplastics morphology tests using Scanning Electron Microscope (SEM). Besides that, it is necessary to improve the mechanical properties and quality of bioplastic.

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